ASSESSMENT OF SPONTANEOUS HEATING SUSCEPTIBILITY OF SOME INDIAN COALS USING EXPERIMENTAL TECHNIQUES

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Bachelor of Technology

In

Mining Engineering

By

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DEPARTMENT OF MINING ENGINEERING
NATIONAL INSTITUTE OF TECHNOLOGY
ROURKELA-769008
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CERTIFICATE

This is to certify that the thesis entitled "**Assessment of Spontaneous Heating Susceptibility of Indian Coals using Experimental Techniques"** submitted by Sri Anujit Bagchi (Roll No. 10605013) in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Mining Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in this thesis has not formed the basis for the award of any Degree or Diploma or similar title of any University or Institution.

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I can‘t represent my words to speak of the inspirations from my mother and sister. I find words inadequate to express any form of acknowledgement to my grandfather who has been an emblem of blessings in all my steps of life.

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Date: **Anujit Bagchi**

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ABSTRACT

INTRODUCTION

Coal mine fire is a major problem worldwide and has been a great concern both for the industry and researchers in this field. Majority of fires existing today in different coalfields are mainly due to spontaneous combustion of coal. Most mine fires start very small and gradually expand in size, thereby, causing loss of natural property, serious environmental pollution of land, water and air and considerable economic losses to the organisation. Although much research has been done on the subject, a proper assessment of the spontaneous heating susceptibility of coal needs to be done so that mine operators are notified well in advance and plan the working properly. Therefore, the determination of susceptibility of coals to spontaneous heating and their classification is essential to plan the production activities and optimise coal mine production within the incubation period.

EXPERIMENTAL INVESTIGATION

To carry out this study, coal samples were collected from the field using channel sampling. Samples from many seams from different coal mines were collected, sealed in air tight containers and carefully brought to the laboratory for further preparation. The collected samples were subject to coning, quartering and grinding. They were then screened to a size of -212 μ and placed inside small air-tight bags. Various field visits were also made to know about the actual conditions that exist in the working conditions and which could be included in the study, during the analyses.

The Proximate analysis for determination of ash, volatile matter and moisture Content was performed to determine the intrinsic properties of coal. Gross calorific values of coals were found out using Bomb Calorimeter. The results of proximate analysis and calorific value are as presented in Table1. The spontaneous heating susceptibility tendency of the coal samples were determined using crossing point temperature (CPT), differential thermal analysis (DTA) and wet oxidation potential difference. The results of these analyses are presented in Table 2. Correlation between the intrinsic properties and susceptibility indices were also carried out.

Table 1: Intrinsic properties of coal samples

Sl. No.	Sample	Proximate Analysis				Gross Calorific Value, GCV (Kcal/kg)
		Ash Content, A (%)	Moisture, M (%)	Volatile Matter, VM (%)	Fixed Carbon, FC (%)	
1.	MCL1	45.508	8.463	25	21.029	3130.98
2.	MCL2	30.95	11.556	27.668	29.826	4023.58
3.	MCL3	15.35	7.952	29.311	47.387	5336.22
4.	MCL4	22.815	6.327	27.904	42.954	5349.685
5.	MCL5	26.336	11.012	26.418	36.234	4798.54
6.	MCL6	22.8	8.346	29.5	39.354	5382.14
7.	MCL7	27.186	6.101	29.944	36.769	4797.98
8.	MCL8	44.02	4.48	25.01	26.49	3738.894
9.	MCL9	52.3	2.4	23.27	22.03	4874.99
10.	MCL10	12.35	14.5	31.97	41.18	5733.5
11.	MCL11	22.88	8.97	29.49	38.66	3947.94
12.	MCL12	7.68	9.59	28.93	53.8	4893.83
13.	MCL13	38.46	11.13	25.19	25.22	3806.58
14.	SCCL1	11.993	4.287	35.159	48.561	6060.54
15.	SCCL2	26.336	2.313	29.72	41.631	5047.92
16.	SCCL3	15.45	2.149	38.704	43.697	6062.55
17.	SCCL4	17.55	4.693	30.641	47.116	5622.7
18.	SCCL5	17.682	6.319	26.311	49.688	5806.8
19.	SCCL6	16.933	2.781	33.219	47.067	6031.3
20.	SCCL7	13.606	2.923	40.677	42.794	6027.46
21.	SCCL8	10.55	5.481	34.619	49.35	6693.61
22.	SCCL9	18.781	4.98	20.007	56.232	4142.48

Table 2: Results of different susceptibility indices of coal samples

Sl. No.	Sample No.	DTA Thermogram Analysis				Crossing Point Temperature, CPT (^{0}C)	Wet Oxidation Potential Difference, (mv)
		Onset Temp., T_c (^{0}C)	Slope				
			II A	II B	II		
1.	MCL1	179.57	0.0747	0.1515	0.0979	154	140
2.	MCL2	164.8	0.1228	0.2064	0.1354	162	124
3.	MCL3	166.23	0.1015	0.1866	0.1137	182	134
4.	MCL4	166.22	0.1205	0.231	0.1364	170	119
5.	MCL5	157.49	0.0958	0.1619	0.1084	167	119
6.	MCL6	170.9	0.0308	0.0929	0.0499	163	121
7.	MCL7	140.73	0.1008	0.1729	0.1182	155	111
8.	MCL8	155.93	0.0122	0.0605	0.0211	169	102
9.	MCL9	147.29	0.016	0.0753	0.0288	174	103
10.	MCL10	158.46	0.0145	0.0743	0.0222	181	51
11.	MCL11	153.34	0.0177	0.066	0.0273	175	48
12.	MCL12	124.82	0.0229	0.0681	0.0295	166	54
13.	MCL13	171.87	0.0167	0.0485	0.0216	169	99
14.	SCCL1	144.98	0.0533	0.078	0.0565	157	107
15.	SCCL2	176.31	0.0615	0.1392	0.0836	161	108
16.	SCCL3	153.78	0.056	0.1245	0.0744	160	100
17.	SCCL4	141.1	0.0445	0.0932	0.0562	167	115
18.	SCCL5	122.14	0.1021	0.1076	0.0885	180	115
19.	SCCL6	126.72	0.0712	0.0909	0.0732	161	105
20.	SCCL7	159.19	0.0272	0.0605	0.0373	170	104
21.	SCCL8	127.67	0.0473	0.079	0.056	183	110
22.	SCCL9	159.79	0.0334	0.012	0.0128	170	120

CONCLUSION

It was seen that a good number of samples showed positive correlation and were less susceptible to spontaneous heating. However, reproducibility of experimental results of Crossing Point Temperature and Bomb Calorimeter Method posed a major problem in the study.

REFERENCES

Mahadevan, V., Ramlu, M. A., 1985, Fire risk rating of coal mines due to spontaneous heating. Journal of Mines, Metals and Fuels, August, 357-362.

Banerjee, S. C., Nandy, D. K. , Banerjee, D. D., Chakravorty, R. N., 1972, Classification of coal with respect to their Susceptibility to Spontaneous Combustion, MGMI, July, Vol. 69, No. 2, pp. 15-28.

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Chapter 1

INTRODUCTION

General

Objective

1. INTRODUCTION

1.1 GENERAL

Coal mine fire is a major problem in the coal mining industry worldwide. It has been reported that majority of fires existing today in different coalfields are mainly due to spontaneous combustion of coal. It accounts for a loss of about 300 MT of coal worldwide annually. Spontaneous combustion of coal is the process of self heating resulting eventually in its ignition without the application of external heat. When coal is exposed to air, it absorbs oxygen at the exposed surface. Some fraction of the exposed coal substance absorbs oxygen at a faster rate than others and the oxidation results in the formation of gases. Mainly CO, CO_2, water vapor along with the evolution of heat during the chemical reaction. The process takes place at normal temperature, but it is slow and the heat evolved is not carried away by air. If the rate of dissipation of heat is slow compared with the evolution of heat by oxidation, there is a gradual build up of heat and temperature reaches the ignition point of coal with then catches fire.

Apart from causing accidents, resulting in loss of lives and property, spontaneous heating of coal also cause considerable economic losses to the organization. Development of spontaneous heating in any section/part of a mine creates problems for the workings of other areas. Some of the other problems associated with spontaneous heating are diminution of heating values and coking properties, and in some cases serious environmental pollution. A careful analysis into occurrence of these fires reveals that most of these fires could have been averted if suitable preventive measures have been taken. The first step for taking proper preventive measure is to accurately assess the spontaneous heating susceptibility of coal seams.

Different coal producing countries of the world follow different methods to assess the spontaneous heating tendency of coal by carrying out different experiments in the laboratory, viz. Crossing Point Temperature (CPT) in India, Russian U-index in Russia, Olpinski index in Poland, Adiabatic Calorimetry in U.S.A. etc. Some of the other methods attempted by researchers are Differential Thermal Analysis (Banerjee and Chakravarty, 1967; Gouws and Wade, 1989a), Wet Oxidation method (Singh et al., 1985; Tarafdar and Guha, 1989), Gas Indices studies (Panigrahi and Bhattacharjee, 2004; Singh et al., 2007) etc. However, there is no unanimity among researchers for the adoption of a particular method for the assessment of

spontaneous heating susceptibility of coals. In fact some of the researchers have suggested that a number of methods may be attempted to determine fairly accurately the degree of proneness of a particular coal to spontaneous heating.

1.2. OBJECTIVE

Keeping the above points in mind, the present work has been planned with the following objectives:

- Collection of coal samples from different mines of different coalfields.
- Determination of intrinsic properties of the collected coal samples.
- Determination of spontaneous heating susceptibility by different experiments, viz. crossing point temperature, differential thermal analysis and wet oxidation potential analysis.

Chapter 2

LITERATURE REVIEW

2. LITERATURE REVIEW

The following is the brief review of the work carried out by different researchers to determine the spontaneous heating tendency of coal samples.

Banrjee and Chakravarty (1967) have suggested differential temperature Analysis(DTA) for the study of spontaneous combustion of coal, particularly in classifying coals with respects to their susceptibility to self heating. A standard experimental procedure for carrying out DTA studies had been prescribed by them. Calcined alumina is recommended as inert reference material for DTA experiments. A heating rate of 5^0C/min was advised for such studies. Typical temperatures obtained from various coals are includes in this study to explain self heating phenomenon.

Banerjee (1972) determined the Crossing Point Temperature (CPT) of a number of Indian coal samples following the Crossing Point Temperature method. He observed that coals with crossing points temperatures between 120^0C & 140^0C could be considered to be highly susceptible to spontaneous heating and those above 160^0C are poorly susceptible. The moderately susceptible ones showed values in between the above mentioned.

Nandy et al. (1972) noted the variation in Crossing Point Temperature values with the volatile matter, oxygen percentage and the moisture content of coal. He found that CPT normally decreases with the increase in each of these constituents of coals. But beyond 35% V.M, 9% oxygen, or 4 to 6% moisture content there is not much change in CPT values. In fact, above 4 to 6% moisture content in coal (on as received basis), the CPT values shows a rising trend.

Feng et al (1973) developed a composite liability index using the results of Crossing Point Temperature experiments, called FCC index. This is calculated using the following equation:

$$\text{Liability Index} = \frac{\text{Average heating rate between } 110^0\text{C and } 220^0\text{C}}{\text{Relative ignition temperature}} \times 10$$

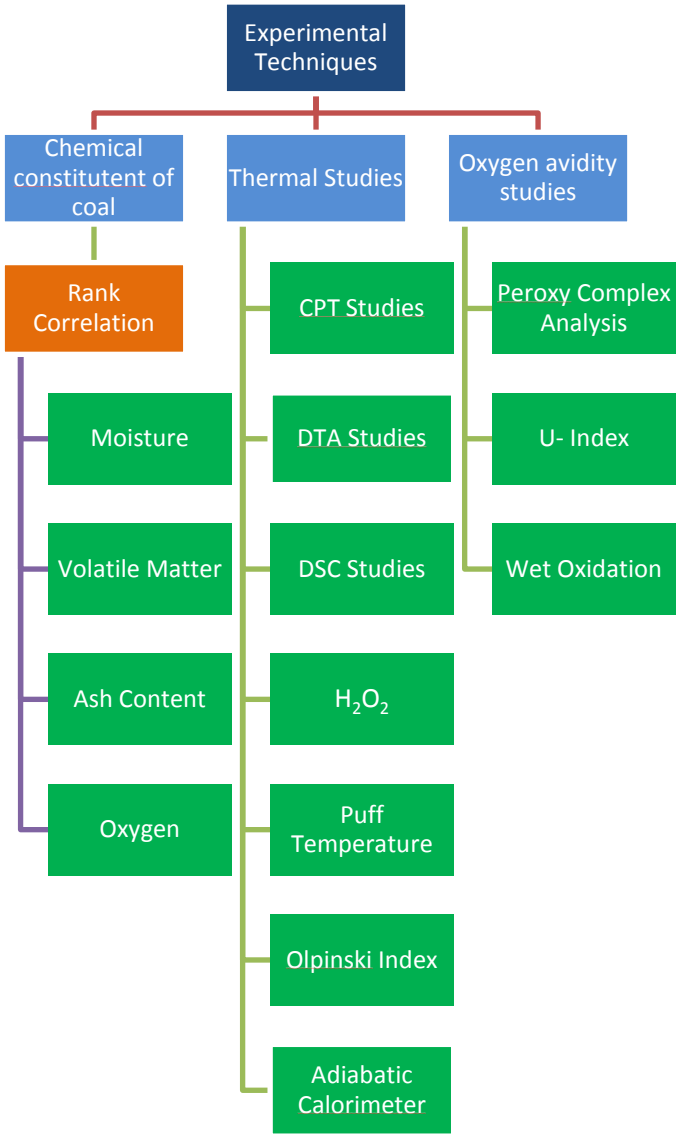
They selected the lower limit for the heating rate at 110^0C in order to ensure that all the moisture had evaporated from the sample. The upper limit of 220^0C was chosen as there would have been little evolution of volatile matter below this temperature.

Mahadevan and Ramlu (1985) objected to the arbitrary selection of temperature ranges in the FCC index and proposed an index known as MR index as:

$$\text{Liability Index} = \frac{\text{Heating rate at the crossing point}}{\text{Time taken to reach the crossing point}} \times \frac{\text{Time taken to reach the inflexion point}}{\text{Average heating rate between the inflexion and crossing point}} \times 10$$

They stated that the reciprocal of their index increases with self heating liability.

Banerjee (1985) studied the different experimental techniques available to assess the spontaneous heating susceptibility of coal and summarised them in the following manner:



Tarafdar and Guha (1989) carried out a preliminary investigation using wet oxidation method and they suggested that a systematic and through study along this lines is required asses the significance of this technique.

Panigrahi et al. (1997) conducted experiments for the determination of Russian U-index. 10 coal samples from Jharia coalfields have been analyzed using this method. The carbon,

hydrogen, nitrogen and sulphur contents for these samples were been determined by using Fenton's method of ultimate analysis. In addition to this, the crossing point temperature of these samples was also determined. Then, attempts were made to correlate the Russian index and CPT of coal samples with its basic constituents viz. carbon, hydrogen and ash contents. It has also been observed that from point of the susceptibility of spontaneous combustion, Russian U-index, shows similar relation with the basic constituents of as the crossing point temperature, which may prove to be a handy method of coal categorization in Indian context.

Thus it could be seen from the above review that there is no universally accepted method or procedure to determine the spontaneous heating tendency of the coal. Keeping this in mind, it is planned to carry out different experiments, viz. crossing point temperature, differential thermal analysis and wet oxidation potential analysis to determine the spontaneous heating tendency of some coal samples in the present dissertation work.

Chapter 3

MECHANISM OF SPONTANEOUS HEATING OF COAL

Mechanism of Spontaneous Heating of Coal

Factors affecting Spontaneous Heating of coal

Theories of Spontaneous Heating

3.0 MECHANISM OF SPONTANEOUS HEATING OF COAL

3.1. MECHANISM OF SPONTANEOUS HEATING

Coal is a heterogeneous and stratified organic rock, with carbon content varying from nearly 70% in the lignite stage (young coal) to 96 % in anthracite (measured coal) with sub-bituminous and bituminous coals lying in between. Coal is constituted of petrographically homogeneous components (macerals) formed from transformation of plants under rigorous extraneous conditions of heat and pressure, as well as biochemical processes.

The interaction of oxygen of coal, like all oxidation reactions, is exothermic. The heat from oxidation normally varies from 2.0 to 4.0 cal/ml of O_2 sorbed at N.T.P. There are other factors that assist in the generation of heat, viz. (a) oxidation of pyrites present in coal, (b) bacterial action, (c) crushing of coal due to earth's movements, and (d) sorption of water vapour in coal. But it is the oxidation of carbonaceous matter in coal that is primarily responsible for the initiation of spontaneous combustion. The exact mechanism of spontaneous heating of coal is still obscure. Unless the chemical constitution of coal is fully established, it would be difficult to clearly understand the mechanism. However, most researchers have recognized it to be essentially a surface combination, the nature of which may be dependent on the oxidation time, temperature and type of coal.

The rate of oxidation increases with the increasing fineness of coal. At low temperature, the rate of oxidation of coal with oxygen of air is proportional to the cube root of its specific internal surface. The oxidizing power of coal increases at first and ultimately decays.

The oxycomplex builds up to a maximum with the increase in time of exposure of coal and then ultimately decays. The loosely bound oxygen constitutes an intermediate stage in the oxidative degradation of coal (at ambient temperature conditions).

Absorption of oxygen by coal takes place at all temperatures. The oxidation of coal is heterogenous in character in which the diffusion of oxygen in the fine pores of the coal and the chemical reactions occurring at the same time influence the rate of reaction. At low temperatures (-80 ^{0}C), the physical adsorption of oxygen in coal was dominant but played a minor role from above 0^0C. Chemical reactions set in at a temperature of -10 ^{0}C leading to the evolution of CO, CO_2, and H_2O between 42-55^{0}C. This shows that chemisorption process takes place in the very early stages of the sorption process.

The oxidation rate decreases from first to hundredth hours at constant temperature by $1/10^{th}$ its value and increases tenfold from a temperature of 30-100^{0}C. The oxidation of coal is slow up to the temperature of about 40^{0}C and thereafter the rate increases 1.8 times for every 10^{0}C rise in temperature. The critical temperature above which the process of oxidation becomes self-sufficient is about 50^{0}C for lignite and about 70-80^{0}C for bituminous coal. The self heating temperature of lignite and sub-bituminous coal were found to as low as 30^{0}C and those of bituminous coals were about 60^{0}C for US coals and for Indian coals the value is about 70^{0}C.

3.2. FACTORS AFFECTING SPONTANEOUS HEATING OF COAL

Spontaneous heating occurs only in certain seams and these susceptible seams are frequently adjacent to seams worked by the same method and subjected to the same tectonics, but which have never been the site of combustion. A few seams have only been susceptible in one area and, again, some seams are more susceptible than others. In this chapter the effect of all the factors on spontaneity of coals are discussed.

Every incident of spontaneous combustion, however small, if not dealt with effectively and efficiently in the early stages can develop into open fires or into an explosion of gas or dust, with devastating results.

The whole phenomenon of the spontaneous combustion as aggregate effects of the situation which may be classified as:-

A) **Seam factors**

1) Rank
2) Petrographic Composition
3) Temperature
4) Available Air
5) Particle Size
6) Moisture
7) Sulphur
8) Effect of Previous Oxidation
9) Physical Properties
10) Heating Due To Earth Movement
11) Bacteria
12) Other Minerals

B) Geological factors

1) Seam Thickness
2) Seam Gradient
3) Caving Characteristics
4) Faulting
5) Coal Outbursts
6) Friability
7) Depth of Cover

C) Mining Factors

1) Mining Methods
2) Rate of Advance
3) Pillar Conditions
4) Roof Condition
5) Crushing
6) Packing
7) Effect of Timber
8) Leakage
9) Multi-Seam Working
10) Coal Losses
11) Main Roads
12) Worked Out Areas
13) Heat from Machines
14) Stowing
15) Ventilating Pressure
16) Change In Humidity

3.2.1. SEAM FACTORS

The rank of the coal depends on the character of the original plant debris from which it was formed and the amount of change that its organic matter has undergone during the period of formation. An increasing carbon content and with its decreasing oxygen content is the most commonly accepted criteria of increasing rank. The higher the rank, e.g. anthracite, the slower is the oxidation rate. Whilst lignite of low rank, oxidizes so rapidly that it is often stated that it can't be stored after mining without ignition.

There are however numerous anomalies to a straight rank order. One part of a seam may be particularly liable to spontaneous heating, a seam of higher rank may prove more troublesome than one of lower rank or even the same in different mines may react differently.

Petrographic composition: Among the petrographic constituents of coal, fusain is the least reactive and durain is more reactive than vitrain.

Temperature: The absorption of oxygen is more rapid as the temperature increases. There is a pronounced temperature coefficient of oxidation and the average rate of oxidation approximately doubles for every rise of 18 degree Fahrenheit.

Available Air: Where there is a small amount of air, the rate of oxidation is very slow and there is no appreciable rise in temperature .Where there are large quantities of air passing over or through the coal, any heat produced will invariably be carried away so that the temperature does not rise and the oxidation rate remains at a low level. However, between these two limits there is a state when the air quantity is sufficiently to promote oxidation but not sufficient to carry away the heat formed, so that there is an accelerating rate of oxidation until ignition occurs.

Particle size: A solid coal face generally presents very little danger of spontaneous combustion, partly due to the small surface area and partly due to the very low permeability of solid coal gases. It is however, generally when coal is shattered in mining, or broken by roof pressure, when falls and faulting occur that spontaneous combustion is likely to take place. It is the small coal that is mainly responsible for the heating. The air passes into the mass oxidizes a little of the coal near the outer surface. They produces a slight rise in temperature, so that as the air penetrates deeper and deeper, it becomes warmer and warmer and although part of its oxygen has been absorbed there is still enough to produce oxidation. Consequently, it is at some distance inside the mass that heating develops most rapidly. It should be noted that a flame is due to the combustion of gas and this requires that a moderately high proportion of oxygen be present. Once the oxidation process has gone beyond the early stages and heat is accumulating, it is only a matter of time before actual ignition takes place.

Moisture: The effect of moisture on spontaneous heating is uncertain. A small quantity seems to assists rather than retard the heating whilst large quantities of moisture retard the heating. However, as in a surface stockpile, alternate drying and wetting of the coal accelerates the heating process.

Sulphur: It was assumed that sulphur in the form of pyrites was the main cause of spontaneous combustion. However, it was later shown that coal even in the absence of sulphides would absorb oxygen and heat spontaneously. However, further research work modified this view and led to the present theory that pyrites plays a subsidiary role in promoting deterioration and spontaneous combustion.

Other minerals: Many other chemicals affect the rate of oxidation to some extent, either accelerating or retarding it. Alkalis can act as accelerators, and borates and calcium chloride as retardant.

3.2.2. GEOLOGICAL FACTORS:

Seam thickness: Where the seam thickness is greater than that which can be completely mined in one part, the area is more susceptible to spontaneous combustion, since the unmined area tends to be subjected to sluggish ventilation flow. It was found that spontaneous combustion was apparently dependent upon the physical factors involved by the thickness of the seam, the methods of working type of ventilation and the friable nature of the coal. Also, in thick seams certain bands within the section can be more liable to spontaneous combustion than others. The thicker the seam, the more difficult it becomes to avoid leaving relatively high risk coal within the goaf area. In some cases it is necessary to mine the waste. However, leaving the low risk coal in the waste invariably tends to be practically impossible. In certain cases, a coal roof and floor way thus be left where,

a) The natural floor or roof tends to be weak

b) The seam is thick, or

c) There are inferior coals over or below the seam.

d) There are inferior coals over or below the seam, in those cases there will undoubtedly be coal in a broken form places where it can heat, and the points of dander presented.

Seam gradient: Flat seams where bord & pillar and long wall methods are used are less susceptible to spontaneous heating. In an inclined seam control of combustion becomes more complex, since convection current resulting from the difference in the temperature must tend to cause air currents in the goaf. In addition, within the extracted areas, flow may be due to buoyancy as a result of the differing densities of the methane, carbon dioxide and nitrogen. Which again may influence the development of spontaneous combustion in the waste, goaf or old working?

Caving characteristics: In the mines where partial extraction is practiced, adequate pillars are left to support the superincumbent strata, and the caving characteristics are generally of little significance. In order to reduce the amount of leakage air flow within the extracted areas, it is desirable for waste to be filled with as fine a material as possible .Thus, this material occupies the greatest volume and fills the void. The type of friable waste and sufficient emphasis should aid on the non- combustible or non-carbonaceous material.

Faulting: Faulted group frequently has an influence on spontaneous combustion. Any grinding action along the fault plane, with the resulting formation of the fine coal, may leads to spontaneous heating. A fault generally slows down the rate of face advance to a safe minimum, with the attendant risk of heat development.

Coal outburst: Coal outbursts usually occur in the harder formation rather than in the softer one and lower coals are more susceptible to spontaneous combustion. However, great care must be taken where there is a possibility of coal outbursts and spontaneous combustion occurring concurrently, as the danger of the products of an outburst i.e. finely powdered coal and /or methane, passing over the site of an active heating are very great.

Coal friability: The more friable the coal is the larger the surface area exposed to oxidation, thus tending to yield more heat per unit volume of coal.

Depth of cover: The depth of cover does not necessarily affect the risk of spontaneous combustion. In general, the greater the depth of cover, the higher the natural strata temperature and thus higher is the base temperature of insight coal. With increase in depth, strata temperature increases with a rate of 40m per ^{0}C.

Geothermal gradient does not directly affect the heating hazards, however, where geothermal gradient are high, the strata temperature in the working is likely to increase more rapidly with increasing cover than where the gradient is low.

3.2.3. MINING FACTORS

Mining methods: An advancing method of mining on the long wall system leaves extracted areas lying between the entries serving the working places .the ventilating pressure differences will encourage air to flow across these areas, with the accompanying risk of combustion. In high-risk situation a retreat system of working is normally preferred. However, the most important advantage gained by adopting a retreating system is lost if an attempt is made to ventilate the waste using a bleeder entry system.

Rate of advance: Within any waste area adjacent to working place there will be air entering the waste, either by the force of ventilating current or deliberately induced by a bleed action. In this area, the rate of flow can be critical. In practice, when a working face is operating normally, any individual piece of coal passes through the zone at a rate equal to the rate of advance of the working place. It is the time taken from entering or leaving the zone that is

critical. If the time is excessive, the oxidation may occur to an unacceptable degree and a glob fire could result.

Pillar Size: Pillar size has a direct influence on the liability to heat. Ideally, pillars should be of a size to avoid crushing. This size depends on the strength of the coal, the coal, the depth of the cover and the influence of other workings with in the vicinity. Increase methane emission is an indication of crushing, around pillars that subsequently spontaneously heated. Generally in coalmines, depending upon the depth of the seam from the surface, size of the pillar is determined. In bord and pillar working at moderate depth, if the pillar size is 30m × 30m center to center, then chances of crushing will be minimized.

Roof condition: Poor roof releases the shock waves to pass though easily and the development of crack increases, so poor roof conditions increases the liability to spontaneous combustion .these falls cavities, which have to be supported and are often filled with timber. Such areas are often sites of localized heating.

Crushing: Crushing is significant in two types of locations: at pillar and rib edges and at worked out areas. Where a pillar is subjected to crush, a situation can develop where leakage paths are created, leading to the flow of air into the coal and in some circumstances, through the solid to affect a more distant zone. Loose coal is usually present in worked out areas, and is produced either by pillar spelling or by roof collapses with associated sluggish ventilation.

Packing: Where, packs are used, in seams, are liable to spontaneous heating. Experiences have shown that they must be of the highest possible quality. It was largely as a result of improvements in gate side packing the incidence of spontaneous combustion is generally reduced in most of the heading in a very short time.

Effect of timber: The timber props left in the waste caused the coal roof to disintegrate and created a saving thus encouraging the spontaneous heating of coal.

Road ways: Road ways in the seam liable to spontaneous heating are areas of concern due to leakage through crushed solid coal. The risk increases with thickness of coal, as this increases the area of coal exposed. The most common points for incidents are junction, air crossing, doors, regulators, connecting roads, obstruction in the roads, old roadways.

Leakage: To create the circumstances in which spontaneous combustion can occur, there must be a supply of oxygen and a situation where a built-up of heat is possible. This can be

brought about by air leaks through fissures in solid coals and result in a shallow seated heating .this situation can occur where leakage paths exits at air crossings, in and around regulators and doors, and other similar locations where leakage there is a high pressure gradient and tendency for air to attempt to flow through solid coals. The leakage through a stopping depends partly on the difference in the pressure between the two sides of the stopping. Nearly all materials available for making the stopping are more or less permeable than others, it is not feasible to reply on making stopping impermeable and if they were completely impermeable, a danger pressure of fire damp could soon accumulate inside them. It should be done in the mind that the leakage trough the porous material , such as that of a stopping or well stowed goof varies in direct proportion to the difference of pressure on the two sides and not in proportion to the square root of the pressure.

Multi seam working: Where a multi seam situation exits both during the first seam and of subsequent seam, condition can arise with spontaneous combustion hazards for the seam currently being worked, and any other seams above or below of it .for example, where a seam has been worked with another un worked seam underlying it, leakage path can be created in to the lower seam, with a consequent risk of heating.

Coal losses: Coal losses that leave remnant coal in worked out areas is a series hazard and most gob fires result from this factor. There is no normal mining system that can guarantee that remnant will never be left in a waste area. Most mining systems result in a significant loss of coal. The resulting situation, in which the coal is likely to be crushed, finely divided and in a location where build-up of heat is possible, must be considered potential hazards.

Worked out areas: Worked out areas which are not sealed by ventilation stoppings are potential sources of spontaneous heating. They are likely to have interruption in the ventilation system as result of roof falls or flour lift leading to deterioration in rib condition and the presence of loose, small crushed coal contributing to potential combustion.

Heat from machines: Normally heat from machine is dissipated within the ventilating air – stream and the temperature rise of the general body of the air are likely to be very small. In some circumstances, the effect of the heat from machines is secondary, in that additional air may have to be circulated and will require a higher ventilating pressure with consequent increase risk of leakage.

Stowing: Stowing is carried out to seal mined out areas completely. It has proved to be an effective method of spontaneous control.

Ventilating pressure: The flow of air in the mines is necessarily associated with pressure difference. This pressure difference is created by the mine fans and the natural ventilation, whist the pressure distribution underground depends upon the resistance of the air ways and distribution of the quantity. Air will try to flow wherever there is a path and a pressure difference, and this means that air will flow along any break or crack in the strata that is open to a pressure difference. A high ventilating pressure differential appears to be an important factor contributing to the spontaneous heating.

Barometric Pressure: It is generally agreed that air may find its way into a sealed-off areas as a result of at least one of the following causes

a) Continuous leakage, resulting from a difference in pressure on the return and intake stopping.
b) Barometric changes
c) Fluctuation in ventilating pressure resulting from the opening of doors and the movement of edges and mine cars.

Humidity: If coal absorbs moisture from the ventilating air it will heat up due to the release of the latent heat of condensation and chemisorptions effects. On the other hand, if the coal loses water by evaporation to the ventilating air, the reverse occurs if there is any imbalance between moisture in the air.

The balance between the complex mining condition that can bring about condensation or evaporation has not fully been researched but it is becoming increasing evident that changes in environmental moisture play a significant role in tipping the balance between adsorption and heating an evaporation and cooling.

3.3 THEORIES OF SPONTANEOUS COMBUSTION OF COAL

3.3.1 Pyrite theory

Heating due to oxidation of pyrites has been known to be a common phenomenon in pyrite mines. Heating of coal can be caused by iron pyrites (only when present in considerable proportion) and in finely powdered and dispersed state in the presence of moisture. The reaction of iron pyrites with oxygen and moisture is exothermic yields products of greater volume than the original pyrite thus opening more pore area for oxygen. The reaction can be given as

$$2Fe\ S_2 + 70_2 + 16H_2O = 2\ H_2SO_4 + 2\ Fe\ S0_4.7\ H_2O + 316\ kcal$$

The oxidation of pyrite during weathering of coal seam may be represented by

$$Fe\ S_2 + 15/4\ O_2 + 9/2\ H_2O = Fe\ O.OH + 2\ H_2SO_4$$

The equations indicate that both oxygen and moisture, two prime weathering agents, contribute to pyrite alteration and the sulphuric acid is formed as a by-product of the alteration. In comparison to dry coals in the presence of moisture, the reactivity of coal is found to be double and if pyrite is in finely dispersed state, it becomes 10 fold. It was seen that pyrite bellow 5% showed negligible effect.

3.3.2 Bacterial Theory

Bacteria were also thought to promote self- ignition of coal. Later investigations proved that that bacteria had little influence on the self – heating tendency of coal. The contribution of heating due to the action of bacteria cannot be completely ruled out. Spontaneous heating observed in haystacks and in wood are known to be mainly due to bacterial action. However, there is no conclusive proof to authenticate or discard this theory.

3.3.3 Phenol Theory

Experiments have shown that phenolic hydroxyls and poly phenols oxidize faster than many other groups. This theory is interesting because it offers a method of determining liability of coal to spontaneous heating.

3.3.4 Electro-chemical Theory

It explains auto-oxidation of coals as oxidation-reduction processes in micro galvanic cells formed by the coal components.

3.3.5 Humidity theory

It states, the quantity of heat liberated by atmospheric oxidation of coal is much less than the quantity of heat required removing water from the coal. If the evaporation of water can be induced at the seat of heating, then the temperature of heating would decrease. When it is recalled that water is an oxidation product of low temperature oxidation of coal, the above scheme well explains other possible sources of CO and CO_2 in low temperature reaction between coal and oxygen. Nordon et al (1979) measured the heat of wetting with water of an Australian low rank bituminous coal with different moisture content. Although dry coal showed a substantial rise of temperature, heat of wetting (6 KJ / Kg) values decreases rapidly with increasing moisture content and heat. For a normal moist coal (65% relative humidity) heat of wetting could cause a temperature rise of only 2° C, which would be unlikely to contribute significantly to self-heating in store coal.

3.3.6 Coal-oxygen Complex Theory

Oxidation of coal is believed to be initiated at native radical sight. Formation of peroxyl radical and hydro peroxides is commonly to be thought to be they mechanism by which oxygen and moisture are initially in corporate into organic matrix. These species may react, rearrange or decomposed to form wide range of oxygen functionality in the matrix or gaseous product.

Chapter 4

EXPERIMENTAL INVESTIGATION

Sample Collection and Preparation

Proximate Analysis

Determination of Calorific Value

Crossing Point Temperature

Differential Thermal Analysis

Wet Oxidation Potential Analysis

4.0 EXPERIMENTAL INVESTIGATION

In order to carry out this study, a number of coal samples were collected from different mines of Mahanadi Coalfields Ltd. (MCL) and Singareni Collieries Company Ltd. (SCCL) covering different seams by channel sampling method. The intrinsic properties of these samples were determined by proximate analysis and bomb calorimetry. The spontaneous heating susceptibility indices were determined by Crossing Point Temperature, Differential Thermal Analysis and Wet Oxidation Potential Analysis Experiments.

4.1 Sample Collection and Preparation

Sampling is the process of collecting the small portion of a whole such that consistence of that portion represents that of a whole It was done by channel sampling method which consisted of cutting channels across the face of exposed ore and collecting the resulting chips, fragments and dust from each channel to make up a sample. First, a coal seam was chosen and the entire length upto a width of 10 cm of it demarcated by chalk was cut to a depth of 10 cm.

4.1.2 Steps of channel sampling

The channel sampling consisted of the following steps:

1. Preparation of surface
2. Demarcation of the channel
3. Cutting the channel
4. Collection the sample
5. Labeling the channel
6. Storing in Sealed bags

1. Preparation the surface: Before cutting the surface, the exposure was cleaned to remove dust, shine, pre-oxidised coal and soluble salts. This may be done by washing the rock thoroughly with a hose or scrubbing with a stiff brush. It was done by chipping off the outer layer of the rock upto a depth of 10 cm along the band within which the sample was to be taken.

2. Demarcation of the channel: When the surface was cleaned, the next step was to mark out the location of the channel by inscribing two parallel lines on the ore body 12-15 cm apart using a chalk or paint.

3. Cutting the channel: Next, the channel was cut using a hand pick (prospectors pick). However a pick is convenient for soft rock like coal and it cannot cope with hard ore bodies. In some mines, the sampling process is speeded by using a light air operated machine drill

equipped with a pointed or chisel shaped bit or a coal drill in case of underground mines where more number of samples are needed to be taken in a single shift.

4. Collection the sample: A sheet of canvas was spread on the floor so as to catch the coal chips as they fall.

5. Labelling the sample: The collected sample was placed in a canvas sack along with an identifying label or tag. It was then brought to the surface.

The sample from different coal fields were brought to the laboratory. The relatively big pieces were crushed to small pieces. Coning and quartering procedure was done to get a representative sample of the entire coal sample. This was then ground and screened (sieving) to a size of - 212μ (micron). Finally, it was kept in sealed packets for further analysis.
Twenty two coal samples were collected by following the above procedure, out of which 13 samples belonged to Mahanadi Coalfields Ltd. (MCL) and the rest nine samples belonged to Singareni Collieries Company Ltd. (SCCL).

4.2 Proximate Analysis

Proximate analysis of coal is a simple means of determining the distribution of products obtained, when the coal sample is heated under specified conditions. It separates the products into four groups: i) moisture; ii) volatile matter consisting of gases and vapours driven off during pyrolysis; iii) fixed carbon, the non volatile fraction of coal; and iv) ash, the inorganic residue remaining after combustion. For proximate analysis, i.e. for the determination of moisture, volatile matter, ash and fixed carbon, the method specified by IS (Indian Standard) 1350 (Part-I) – 1969 was followed.

4.2.1 Determination of Moisture Content

Coal, due to its nature, origin and occurrence, is always associated with some amount of moisture, which is both physically and chemically bound. It is differentiated between external and inherent moisture. When a wet coal is exposed to atmosphere, the external moisture evaporates, but the apparently dry coal still contains some moisture, which can be removed only on heating above 100^0C. External moisture is also called accidental or free moisture, where as inherent moisture is termed as equilibrium or air-dried or hygroscopic moisture. The quantity of external moisture depends mainly on the mode of occurrence and handling of coal, but the air-dried moisture is related to the inherent hygroscopic nature of the coal.

Experimental Procedure

About 1g of finely powdered (-212μ) air-dried coal sample is weighed in a silica crucible and then placed inside an electric hot air oven (Figure 4.1) maintained at 108^{o}C. The crucible with the coal sample was allowed to remain in the oven for 1.5 hours and was then taken out with a pair of tongues, cooled in a desiccator for about 15 minutes and then weighed. The loss in weight was reported as moisture (on percentage basis). The calculation was done as per the following.

$$\% \text{ Moisture } = \frac{Y-Z}{Y-X} \times 100$$

Where X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, g

Z = weight of crucible + coal sample after heating, g

Y - X = weight of coal sample, g

Y- Z = weight of moisture, g

Figure 4.1: Oven for Moisture Content Determination

Figure 4.2: Muffle Furnace for Volatile Matter and Ash Content Determination

4.2.2 Determination of Volatile Matter Content

The loss of mass, corrected for moisture, which results when coal is heated in specified equipment under prescribed conditions, is referred to as volatile matter. The matter lost is composed of materials that form upon the thermal decomposition of the various components of coal. Some of the constituents of coal volatile matter are hydrogen, carbon monoxide, methane, other hydrocarbons, tar vapours, ammonia, some organic sulphur, oxygen

containing compounds and some incombustible gases, such as carbon dioxide and water vapour.

Experimental Procedure

For the determination of volatile matter a special volatile matter silica crucible (38mm height, 25mm external diameter and 22mm internal diameter) was used. The empty volatile matter crucible was weighed. Approximately 1g of coal sample (-212μ size) was weighed in the volatile matter crucible and it was placed inside a muffle furnace maintained at 925^{o}C with the lid covering the crucible. The heating was carried I the muffle furnace (Fig. 4.2) out exactly for seven minutes, after which the crucible was removed, cooled in air, then in a desiccator and weighed again. The calculation was done as per the following.

$$\% \text{ Volatile Matter} = \frac{Y-Z}{Y-X} \times 100 - M$$

Where X = weight of empty crucible, g

Y = weight of crucible + coal sample before heating, g

Z = weight of crucible + coal sample after heating, g

Y -X = weight of coal sample, g

Y- Z = weight of volatile matter + moisture, g

4.2.3 Determination of Ash Content

Coal ash is the residue remaining after the combustion of coal under specified conditions. It does not occur as such in the coal, but is formed as the result of chemical changes that take place in the mineral matter during the combustion process. Ash and mineral matter of coal are therefore not identical.

There are two types of ash forming materials in coal: extraneous and inherent mineral matters. The extraneous mineral matter consists of materials such as calcium, magnesium and ferrous carbonates, pyrite, marcasite, clays, shales, sand and gypsum. The extraneous mineral matter owes its origin to i) the substances which got associated with the decaying vegetable material during its conversion to coal, which is difficult to remove by mechanical methods, and ii) rocks and dirt getting mixed up during mining and handling of coal. Inherent mineral matter represents the inorganic elements combined with organic components of coal. The origin of such materials is probably the plant materials from which the coal was formed.

Ash from inherent mineral matter is insignificant as far as the total quantity of ash is concerned. But Indian coals suffer from the major disadvantage, that the mineral matter content is not only high, but of intimately associated type, due to its drift origin.

Experimental Procedure

The empty crucible was cleaned by heating in a muffle furnace for one hour at 800^0C so that other mineral matter if presents get burnt. It was taken out, cooled to room temperature and the weight is taken. Approximately 1gm of coal sample was weighed in the crucible and placed in a muffle furnace at 450^0C for 30 minutes and the temperature of the furnace was raised to 850^0C for 1hour. The crucible was taken out and placed in a desiccator and weighed.

$$\% \text{ Ash } = \frac{Z-X}{Y-X} \times 100$$

Where X = weight of empty crucible in grams

Y = weight of coal sample + crucible in grams (Before heating)

Z = weight of coal sample + crucible in grams (After heating)

Y - X = weight of coal sample, g

Z - X = weight of ash, g

4.2.4 Determination of Fixed Carbon (FC)

Fixed carbon (FC) is by definition, the mathematical remaining after the determination of moisture, volatile matter and ash. It is, in fact a measure of the solid combustible material in coal after the expulsion of volatile matter. Fixed carbon plus ash represent the approximate yield of coke from coal. The fixed carbon value is determined by subtracting from 100 the resultant summation of moisture, volatile matter and ash, with all percentage on the same moisture reference base.

$$\text{FC} = 100 - (\text{M} + \text{VM} + \text{A})$$

The moisture (M), volatile matter (VM), Ash and Fixed carbon content of coal determined by following the above procedure is presented in Table 4.1.

Table 4.1: Moisture (M), Volatile Matter (VM), Ash (A) and Fixed Carbon (FC) content of coal

Sl. No.	Sample	Proximate Analysis			
		Ash Content, A (%)	Moisture, M (%)	Volatile Matter, VM (%)	Fixed Carbon, FC (%)
1.	MCL1	45.508	8.463	25	21.029
2.	MCL2	30.95	11.556	27.668	29.826
3.	MCL3	15.35	7.952	29.311	47.387
4.	MCL4	22.815	6.327	27.904	42.954
5.	MCL5	26.336	11.012	26.418	36.234
6.	MCL6	22.8	8.346	29.5	39.354
7.	MCL7	27.186	6.101	29.944	36.769
8.	MCL8	44.02	4.48	25.01	26.49
9.	MCL9	52.3	2.4	23.27	22.03
10.	MCL10	12.35	14.5	31.97	41.18
11.	MCL11	22.88	8.97	29.49	38.66
12.	MCL12	7.68	9.59	28.93	53.8
13.	MCL13	38.46	11.13	25.19	25.22
14.	SCCL1	11.993	4.287	35.159	48.561
15.	SCCL2	26.336	2.313	29.72	41.631
16.	SCCL3	15.45	2.149	38.704	43.697
17.	SCCL4	17.55	4.693	30.641	47.116
18.	SCCL5	17.682	6.319	26.311	49.688
19.	SCCL6	16.933	2.781	33.219	47.067
20.	SCCL7	13.606	2.923	40.677	42.794
21.	SCCL8	10.55	5.481	34.619	49.35
22.	SCCL9	18.781	4.98	20.007	56.232

4.3. Bomb Calorimetry

The energy value of coal or calorific value is the amount of potential energy in coal that can be converted into actual heating ability. The value can be calculated and compared with different grades of coal or even other materials. Materials of different grades will produce differing amounts of heat for a given mass. The calorific value of coal is usually determined by the bomb calorimeter method.

Bomb Calorimeter

Bomb calorimeter consists of a stout cylindrical chamber known as bomb (Fig. 4.4) of stainless steel. This chamber is fitted with an air tight cover which can be screwed on the chamber. The cover has three terminals; two for sparking and one for the entry of oxygen. After forcing the oxygen into the chamber the passage can be blocked by screwing in the third terminal.

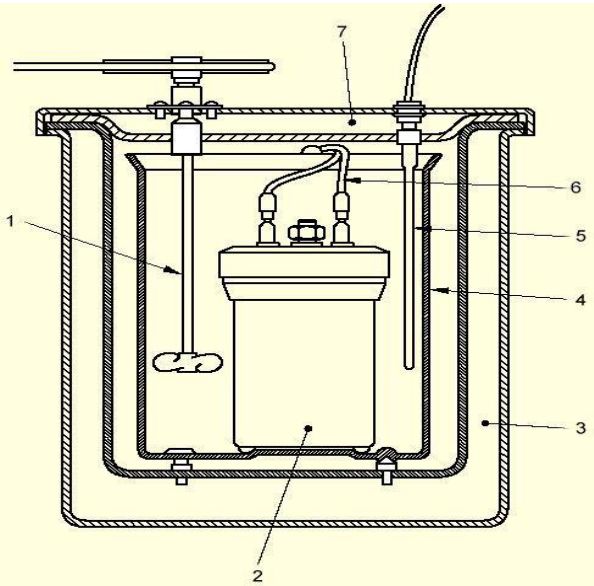


Fig 4.3: Schematic layout of Bomb Calorimeter

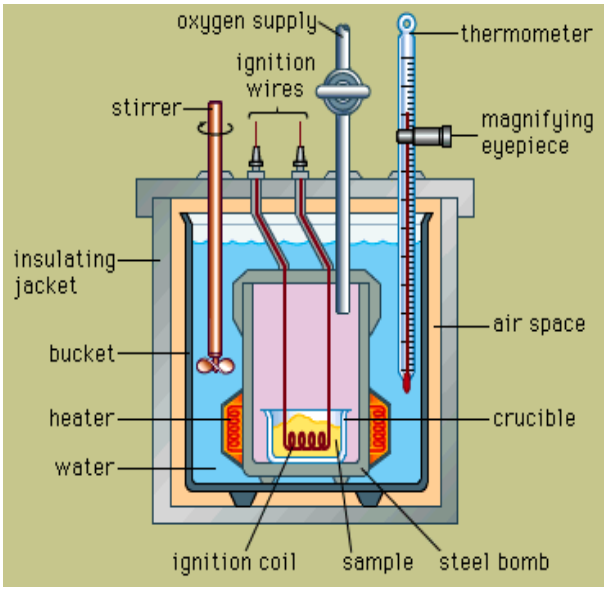


Fig 4.4: Structure of the Bomb

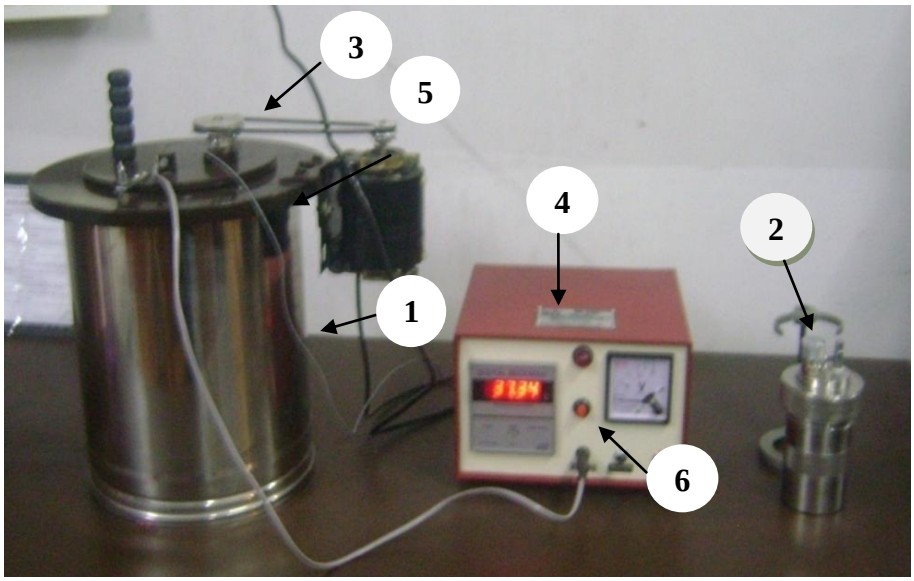
1) Stirrer
2) Calorimeter bomb
3) Jacket
4) Calorimeter vessel
5) Platinum Resistance Thermometer (PRT)
6) Ignition lead
7) Jacket

On the other side of the cover, there are two bent rods connected to two terminals. The bent rods have small holes through which two fuse wires are connected. There is the provision for putting the crucible containing the pellet tied to the fuse wire by means of a cotton thread. This whole set-up is placed in a bigger vessel containing a known quantity of water in it. The vessel is jacketed to minimize the heat loss by radiation. A stirrer is used for stirring the water in the bigger vessel. There is a provision for inserting the thermometer.

Experimental Procedure

Approximately about 1gram of -212μ size air dried coal sample is taken by weighing in a balance. A pellet is made with the coal and weighed. The calorimeter cover is taken and about 10 cc of distilled water is poured into it. The pellet in the crucible is brought in contact with the fuse wire by means of a thread. The cover is then tightened. Oxygen is

then admitted into the calorimeter at a pressure of about 25 atmospheres. 2 liters of water is put into the bigger vessel. The thermometer is inserted into the pocket. Necessary electrical connections are made and stirrer is adjusted in the corrected position. The stirring is done gently for five minutes. The initial temperature reading is then taken. The bomb is now fired. Sparking and combustion of coal take place in the calorimeter. The maximum reached temperature is then noted.



1. Calorimeter vessel
2. Bomb
3. Stirrer
4. Read out unit
5. Cover
6. Firing Cable

Fig. 4.5: Experimental Set up of Bomb Calorimeter

The bomb is removed and the pressure released. The bomb interior is examined for unburnt or sooty deposits. If such material is found, the test is discarded.

The GCV (Gross Calorific Value) is calculated as, GCV = $\frac{2366.5 \times \Delta T}{m}$

where ΔT = Difference between Initial and Final Temperatures and m- the mass of the pellet

The constant 2366.5 is the specific heat capacity of water in kcal/^{o}C. The results of bomb calorimetry are presented in Table 4.2.

Table 4.2: Gross Calorific Values of the Coal Samples

Sl. No.	Sample	Gross Calorific Value, GCV (kcal/kg)
1.	MCL1	3130.98
2.	MCL2	4023.58
3.	MCL3	5336.22
4.	MCL4	5349.685
5.	MCL5	4798.54
6.	MCL6	5382.14
7.	MCL7	4797.98
8.	MCL8	3738.894
9.	MCL9	4874.99
10.	MCL10	5733.5
11.	MCL11	3947.94
12.	MCL12	4893.83
13.	MCL13	3806.58
14.	SCCL1	6060.54
15.	SCCL2	5047.92
16.	SCCL3	6062.55
17.	SCCL4	5622.7
18.	SCCL5	5806.8
19.	SCCL6	6031.3
20.	SCCL7	6027.46
21.	SCCL8	6693.61
22.	SCCL9	4142.48

4.4. Crossing Point Temperature (CPT)

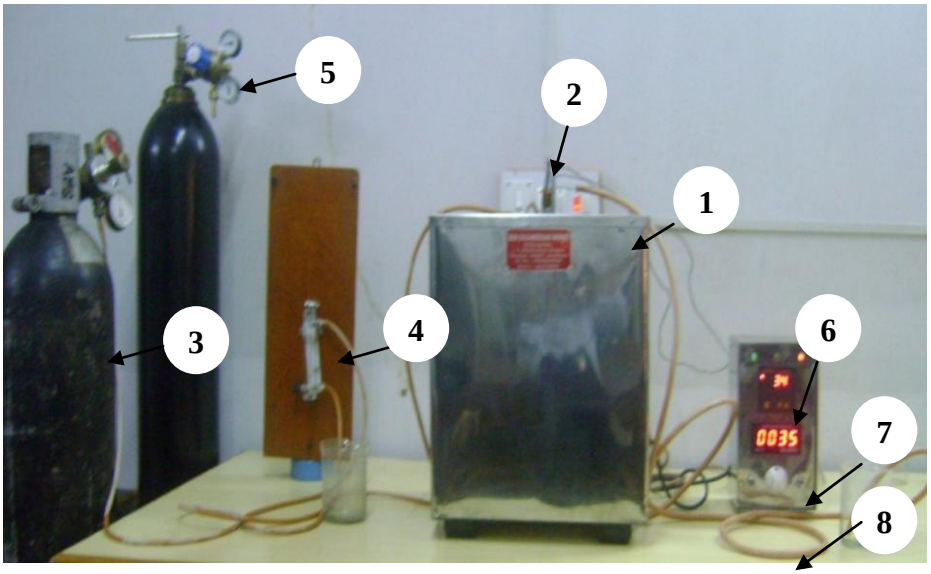
This method envisages heating of coal sample in an oxidizing atmosphere at a definite programmed rate of temperature rise. The lowest temperature at which the exothermic rate in coal bed is self-propagating is termed as Crossing Point Temperature **(**CPT). In other words, crossing point temperature is the temperature at which the temperature of coal in the reaction tube coincides with the bath temperature.

The setup for the determination of crossing point temperature (CPT) of coal consisted of following (Figure: 4.6)

(1) Vertical tubular furnace.

(2) Glass reaction tube. The reaction tube has spiralling glass tube of 6mm internal diameter around it which is connected to the bottom (inside) of the reaction tube for air inlet and a small out-let tube at the top acts as air/gas outlet.

(3) Flow meter and pressure flow control valves.



1. Tubular Furnace
2. Reaction Tube
3. Oxygen Cylinder
4. Flow Meter
5. Pressure Control Valves
6. Bath Temperature
7. Coal Temperature
8. Temperature control valve

Fig 4.6: Experimental Setup of Crossing Point Temperature CPT Apparatus

Procedure

20gm sample of size -212μ was placed in the reaction tube with glass wool at the bottom most position. The tube was then lightly tapped a fixed number of times to achieve uniform packing density of the samples. The reaction tube was then placed in the furnace and a chromel-alumel thermocouple is inserted at the centre of the sample. The furnace was switched on and simultaneously air is allowed to pass through the sample, with an average rate heating of 5^0C per minute and at 80ml/ min. The temperature of the furnace (bath) and

the coal sample was recorded at every five minute interval till the temperature of coal crossed over and went beyond the furnace temperature. From CPT values, the spontaneous heating risk is determined as given in Table 4.3. The results are presented in Table 4.4.

Table 4.3: Risk rating criteria based on CPT values

CPT (^{0}C)	Risk
120-140	Highly liable to self heating
140-160	Moderately liable to self heating
160-180	Less liable to self heating

Table 4.4: Crossing Point Temperature of the Coal Samples

Sl. No.	Sample	Crossing Point Temperature, CPT (^{0}C)
1.	MCL1	154
2.	MCL2	162
3.	MCL3	182
4.	MCL4	170
5.	MCL5	167
6.	MCL6	163
7.	MCL7	155
8.	MCL8	169
9.	MCL9	174
10.	MCL10	181
11.	MCL11	175
12.	MCL12	166
13.	MCL13	169
14.	SCCL1	157
15.	SCCL2	161
16.	SCCL3	160
17.	SCCL4	167
18.	SCCL5	180
19.	SCCL6	161
20.	SCCL7	170
21.	SCCL8	183
22.	SCCL9	170

4.5. Differential Thermal Analysis (DTA)

Differential thermal analysis involves heating a small test specimen at a constant rate and continuously recording the instantaneous temperature difference (ΔT) between it and as identically heated inert reference material as a function of sample temperature (T). The resultant thermogram, a record of ΔT against T, with its characteristics heat changes and intensities, depicts the physical or chemical changes of the material at that particular temperature and is characteristic of that material used.

Experimental Procedure

The experiment was carried out by a Differential Thermal Analyser. The standardised parameters suggested by Banerjee and Chakravorty (1967) were followed while performing the experiments.

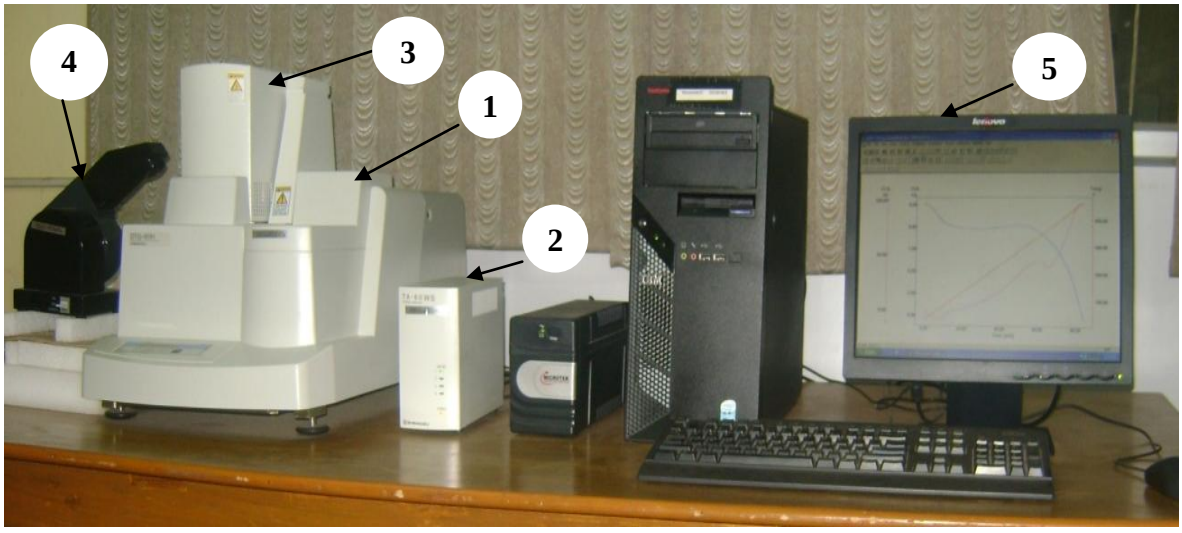


Fig. 4.7: Experimental Set up of Differential Thermal Analysis Apparatus

1) Differential Thermal Analyser (DTA- 60H)
2) TA – 60 WS Collection Monitor
3) Tubular Furnace
4) Blower
5) Computer for Data Processing

The crucible for sample and reference was put in position on the ceramic post. 6-10 mg of -212μ size coal sample was weighed in the crucible and put onto the sample holder. Oxidising atmosphere was maintained by keeping the coal sample exposed to air. Alpha alumina powder was used as the reference material. The tubular furnace was then lowered. The software was programmed to run until 450^0C at a rate of 5^0C per minute. After the heating is terminated,

thermogram is obtained for the sample. A schematic layout of DTA instrument is depicted in Fig. 4.8. The DTA thermograms of all the samples are as presented in Figures 4.9 to 4.30

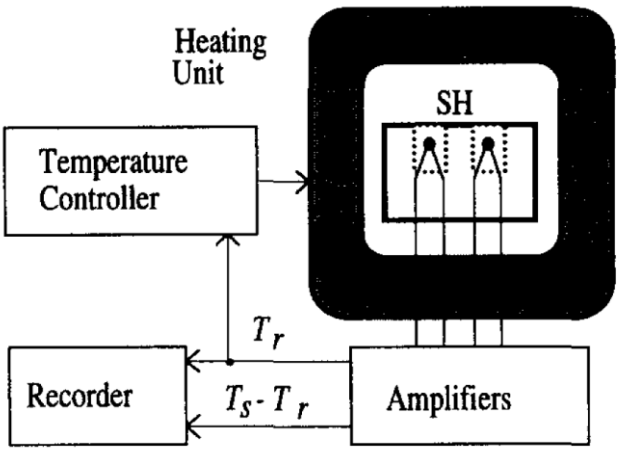


Fig. 4.8: A schematic layout of DTA Instrument

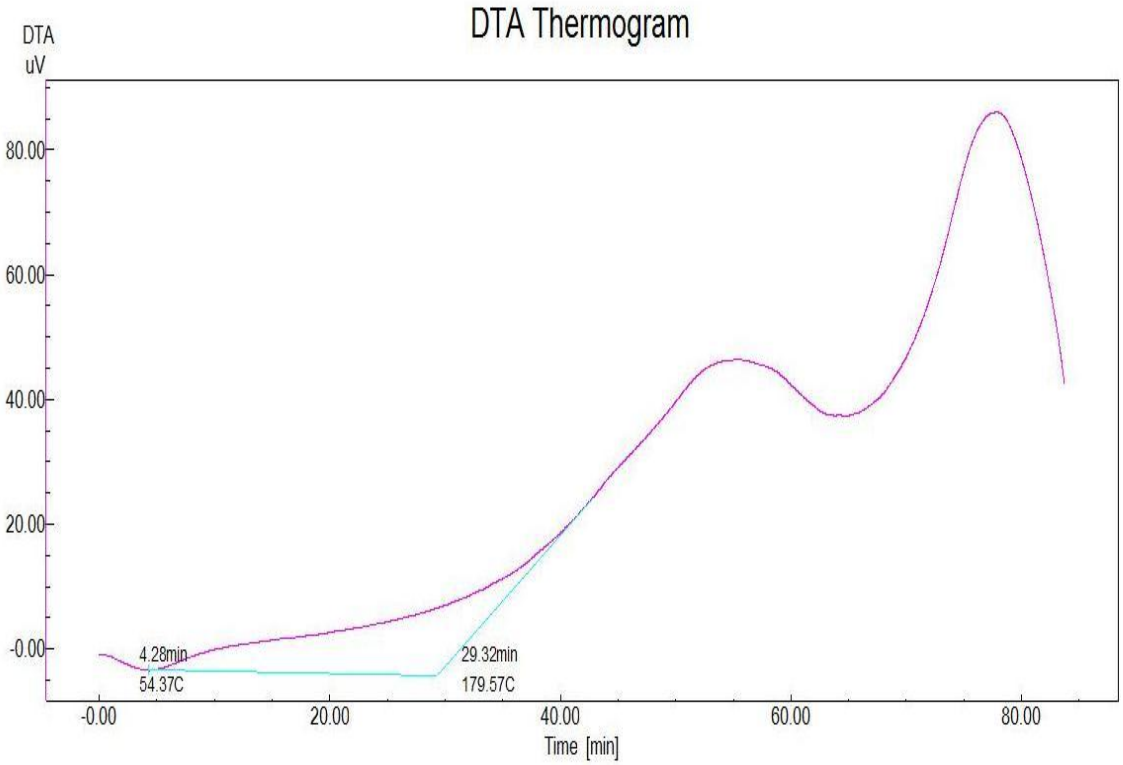


Fig. 4.9: DTA Thermogram for Sample MCL 1

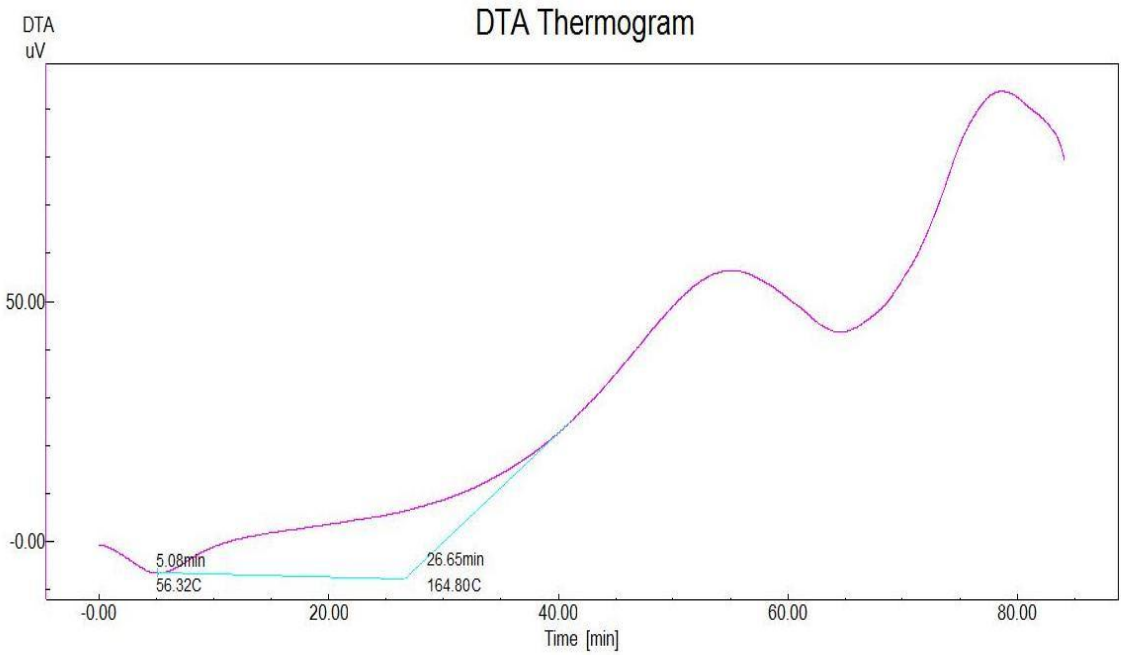


Fig. 4.10: DTA Thermogram for Sample MCL 2

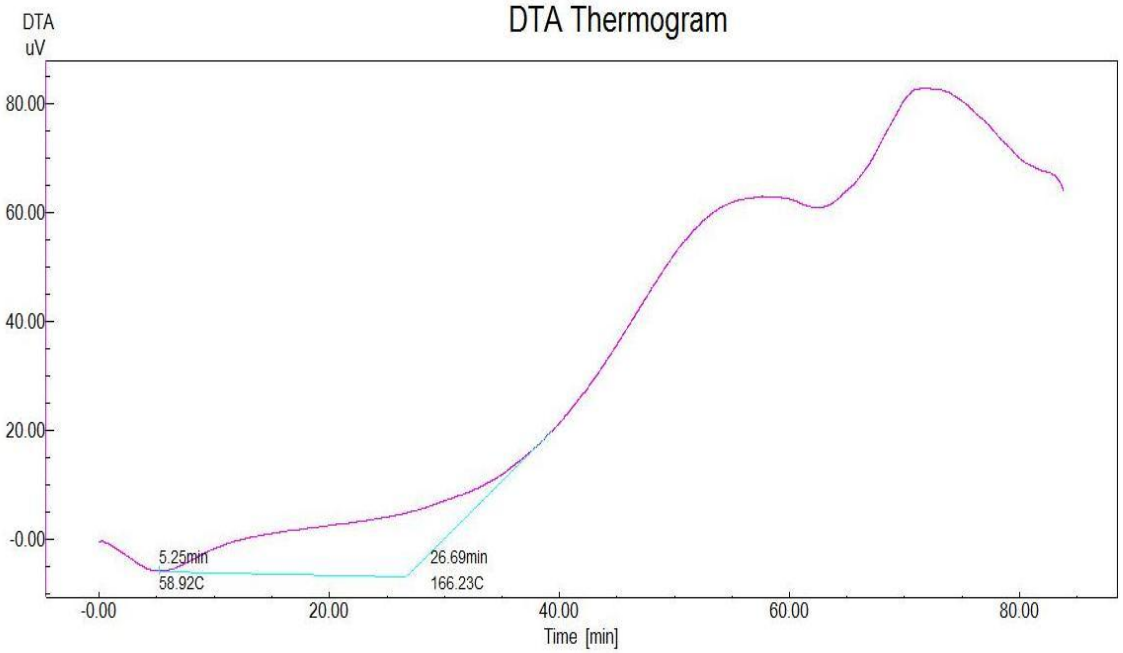


Fig. 4.11: DTA Thermogram for Sample MCL 3

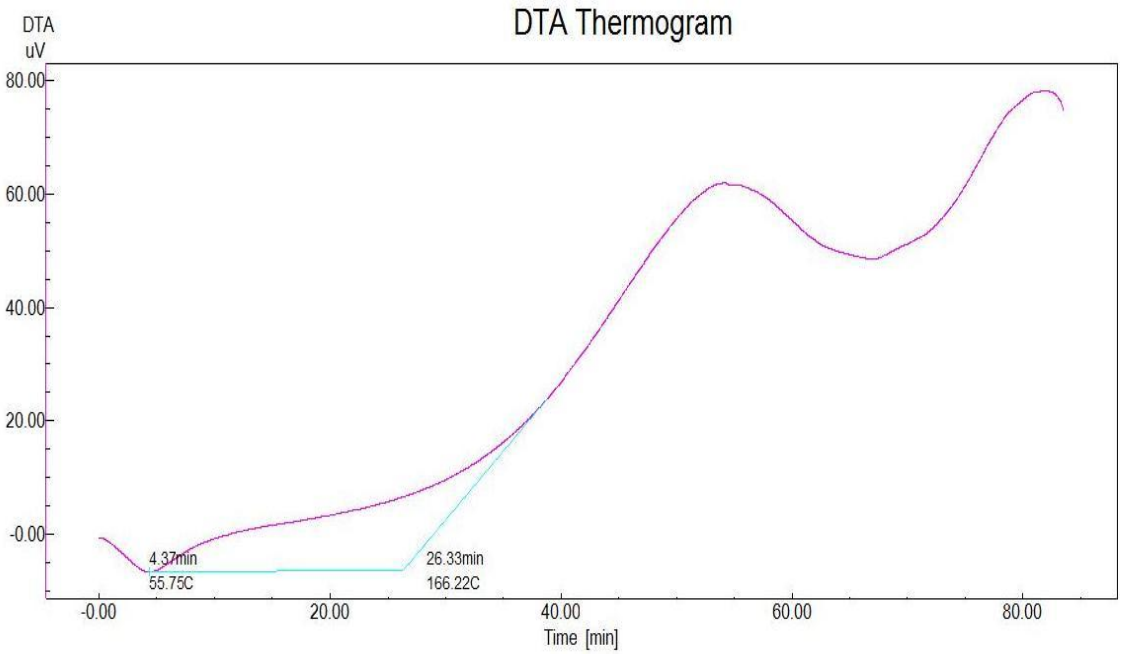


Fig. 4.12: DTA Thermogram for Sample MCL 4

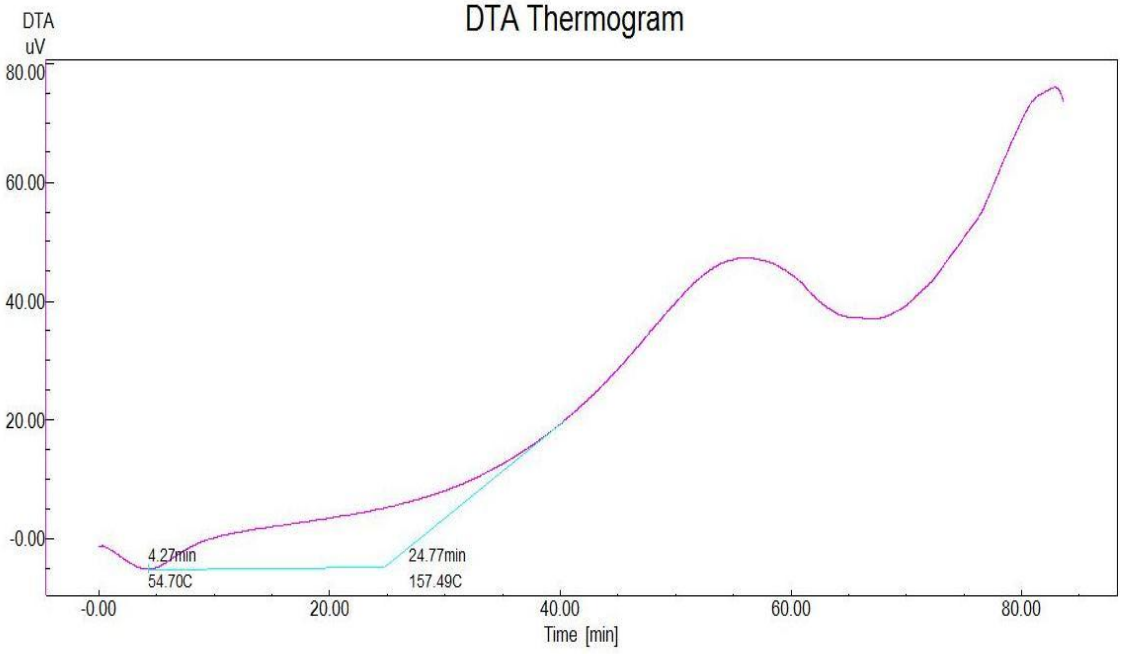


Fig. 4.13: DTA Thermogram for Sample MCL 5

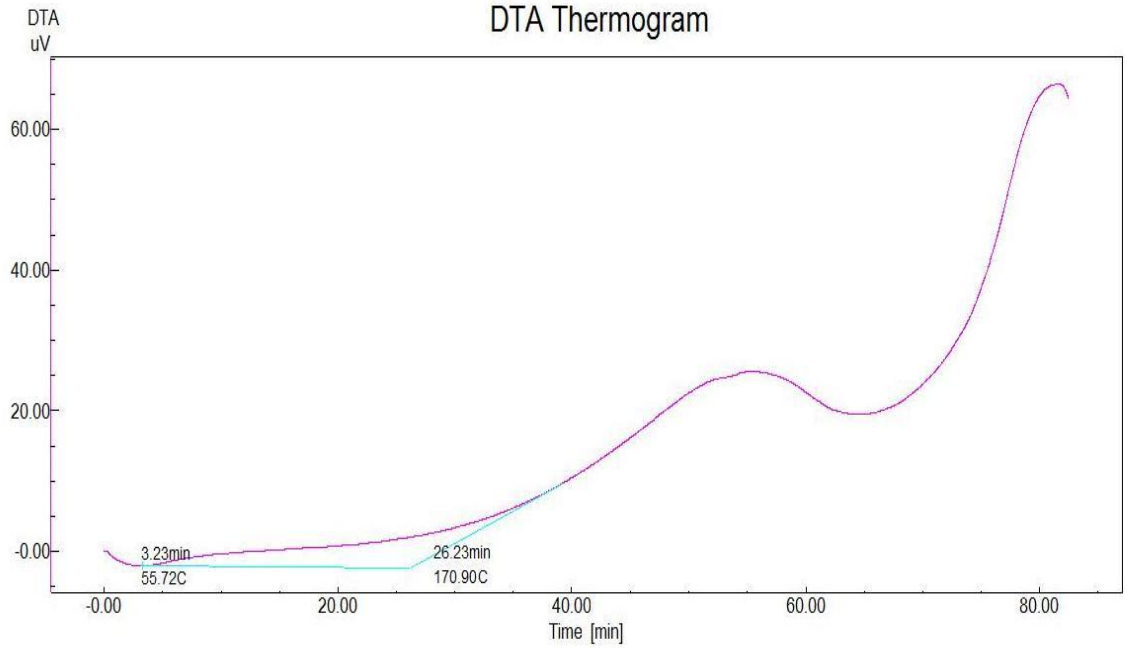


Fig. 4.14: DTA Thermogram for Sample MCL 6

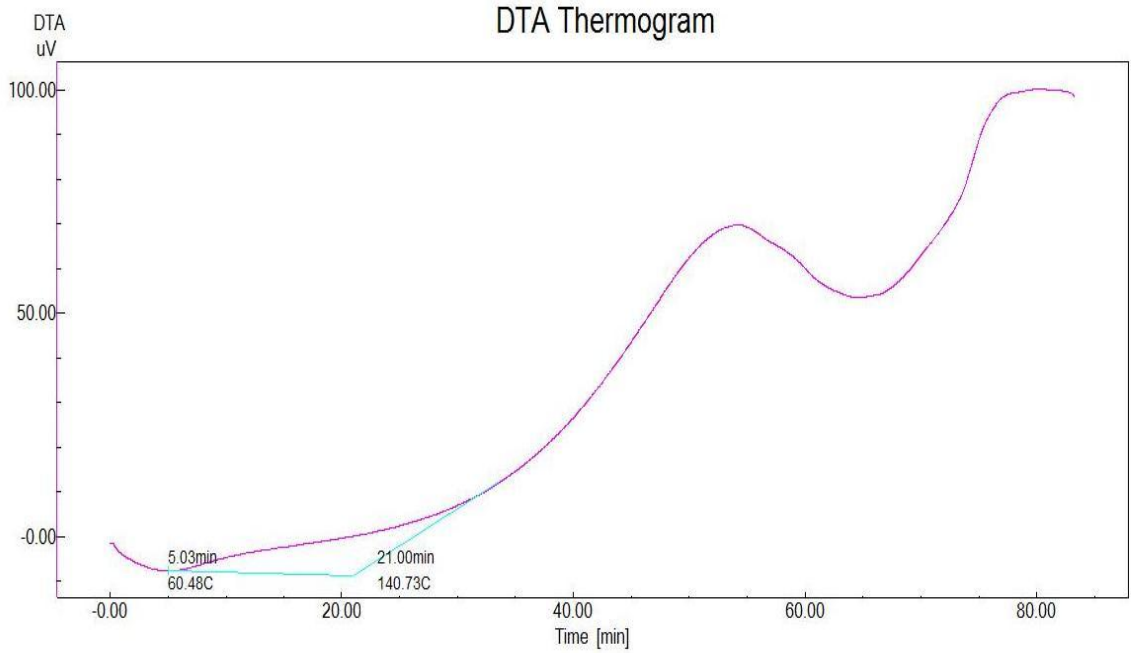


Fig. 4.15: DTA Thermogram for Sample MCL 7

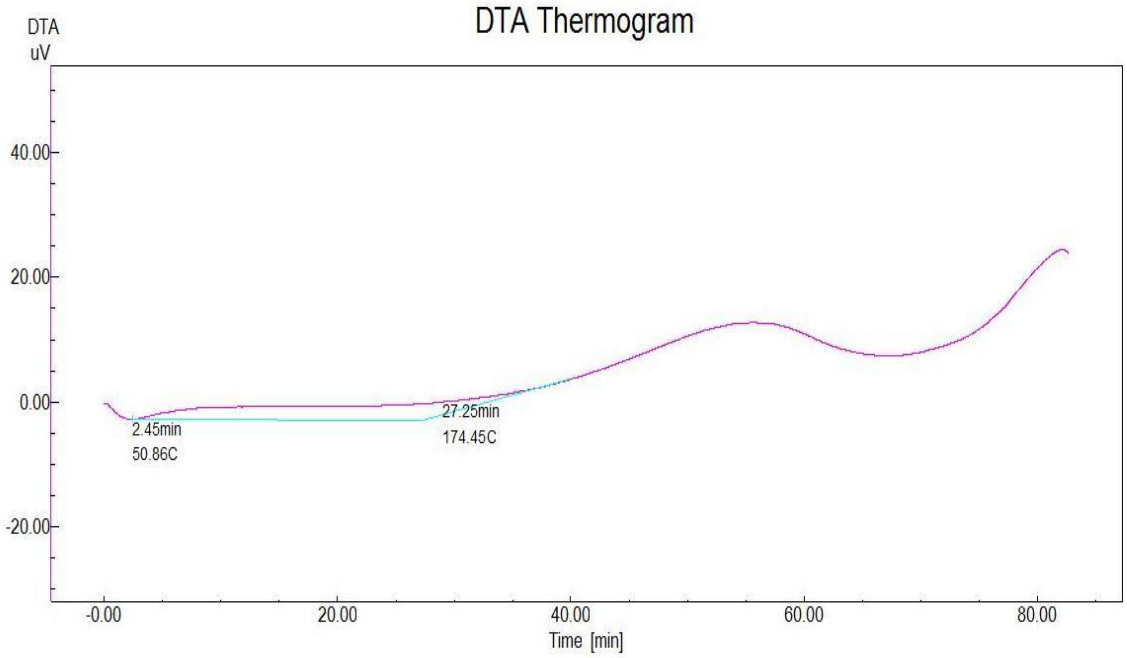


Fig. 4.16: DTA Thermogram for Sample MCL 8

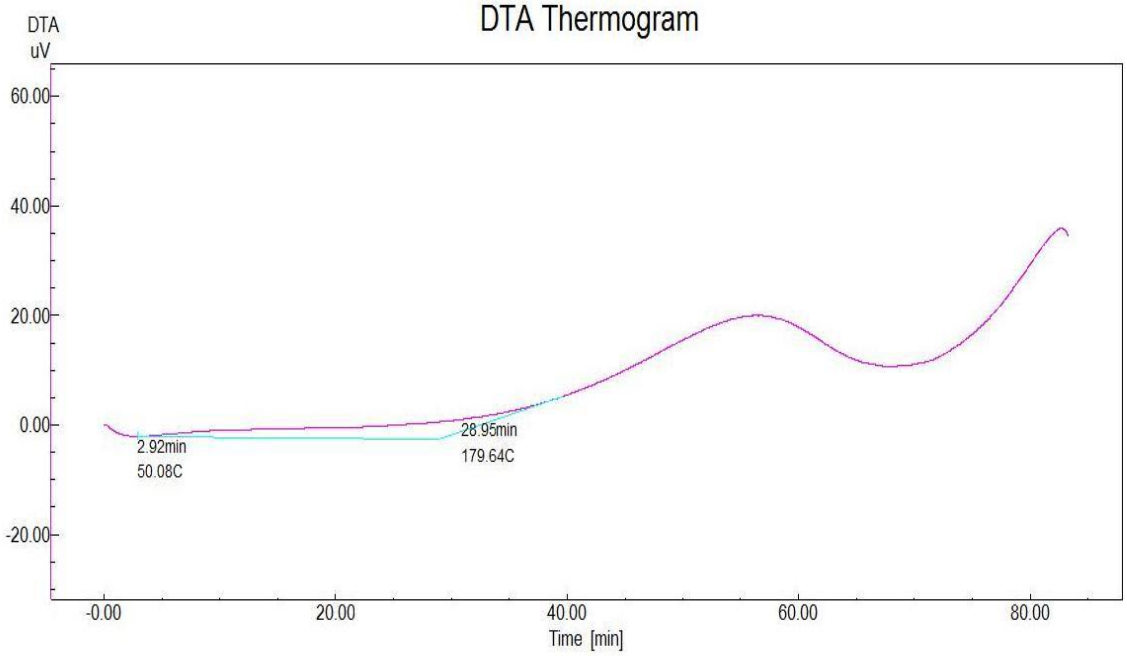


Fig. 4.17: DTA Thermogram for Sample MCL 9

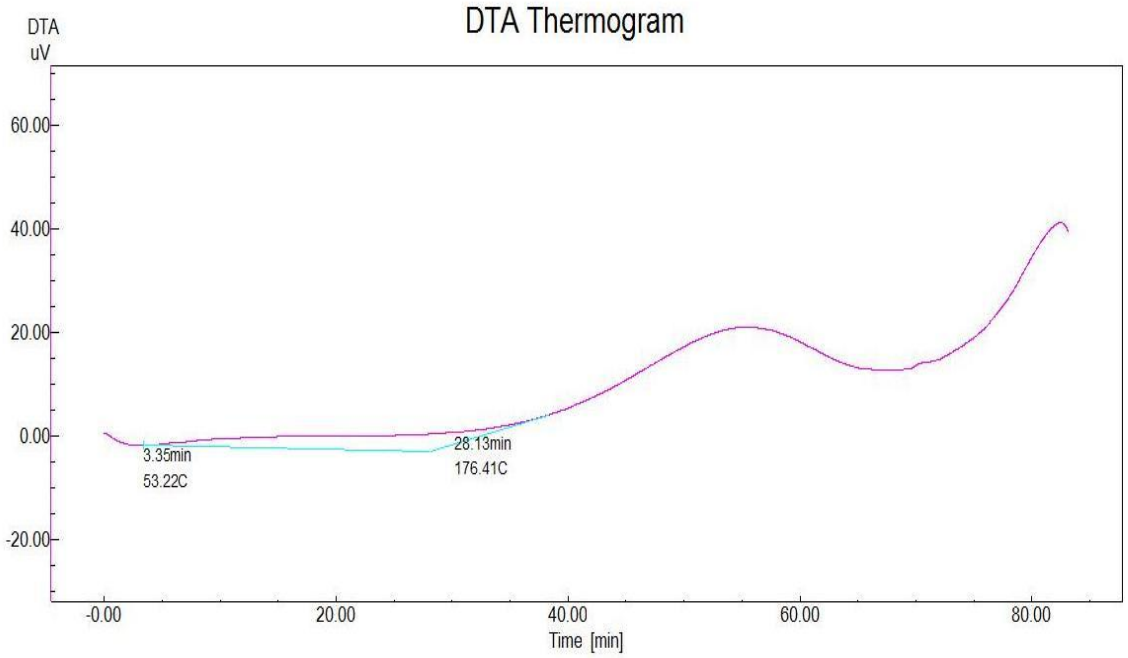


Fig. 4.18: DTA Thermogram for Sample MCL 10

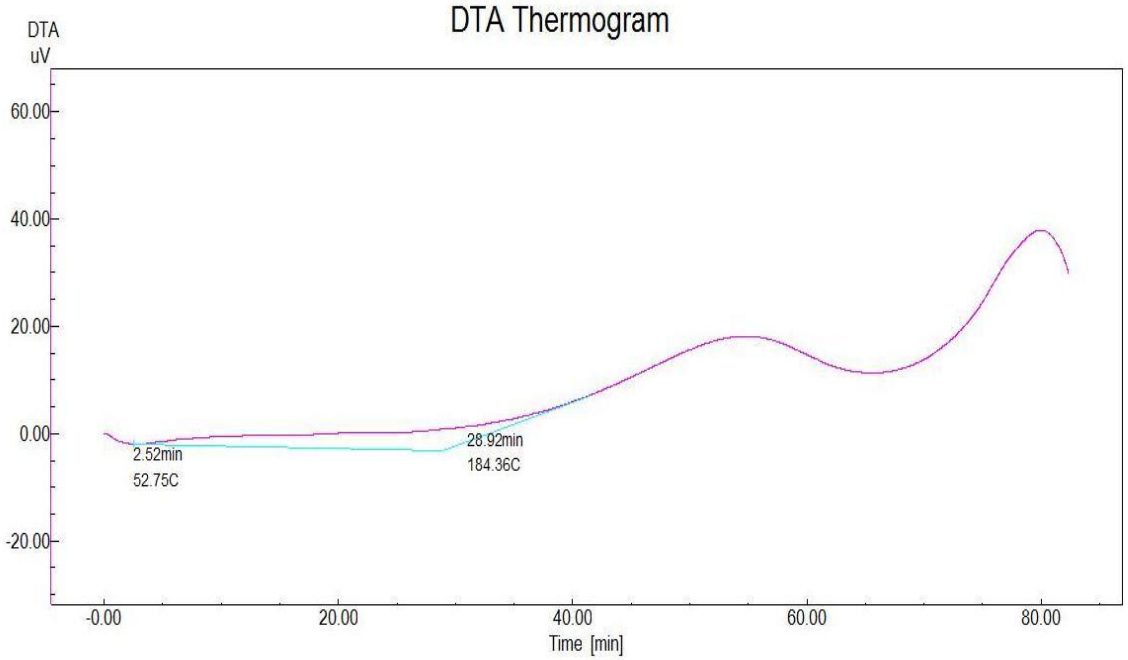


Fig. 4.19: DTA Thermogram for Sample MCL 11

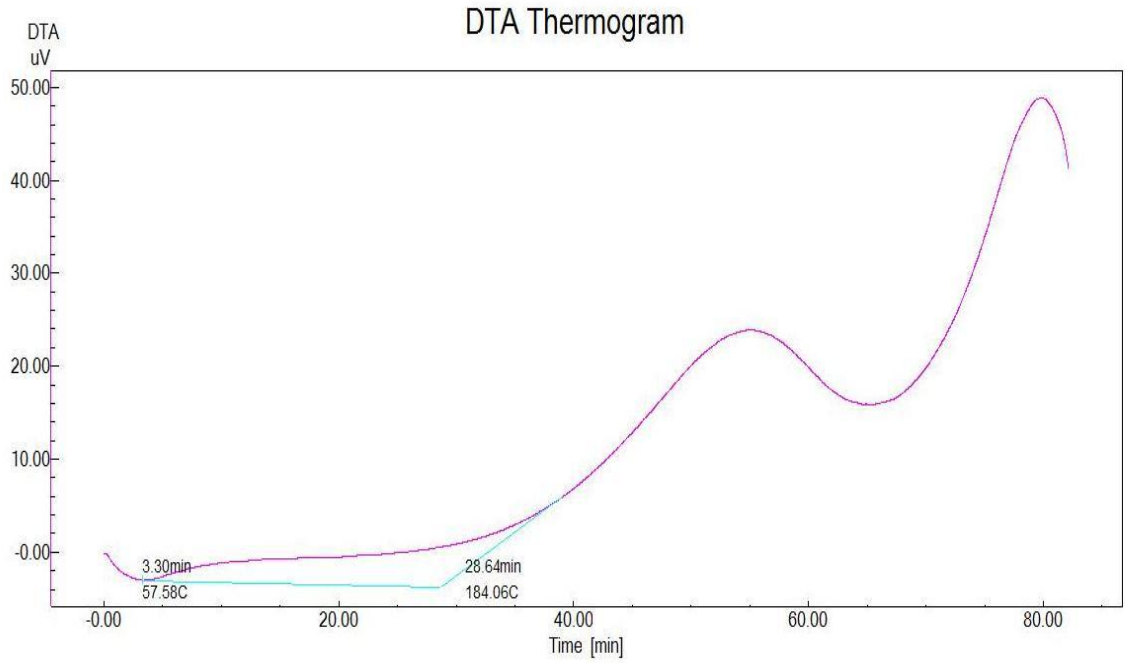


Fig. 4.20: DTA Thermogram for Sample MCL 12

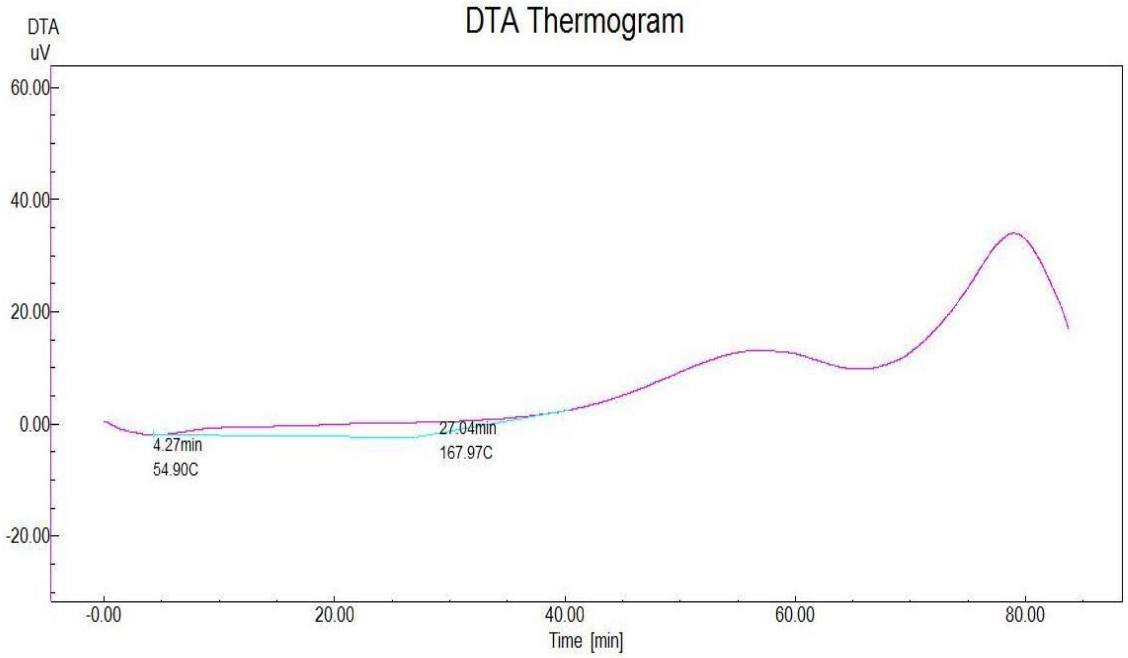


Fig. 4.21: DTA Thermogram for Sample MCL 13

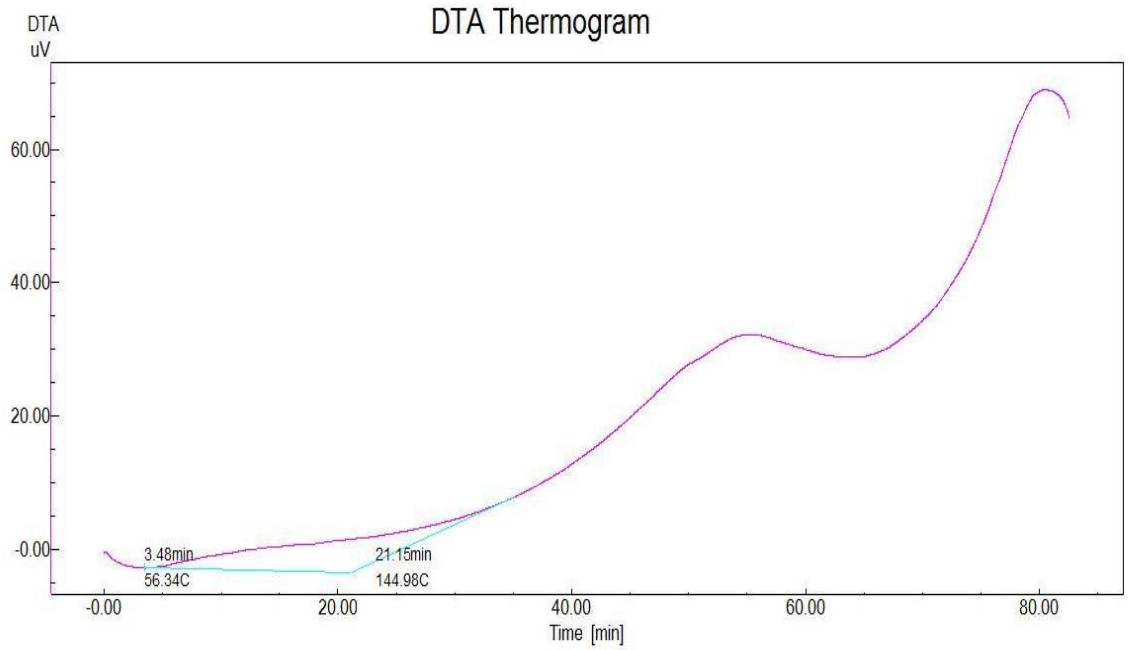


Fig. 4.22: DTA Thermogram for Sample SCCL 1

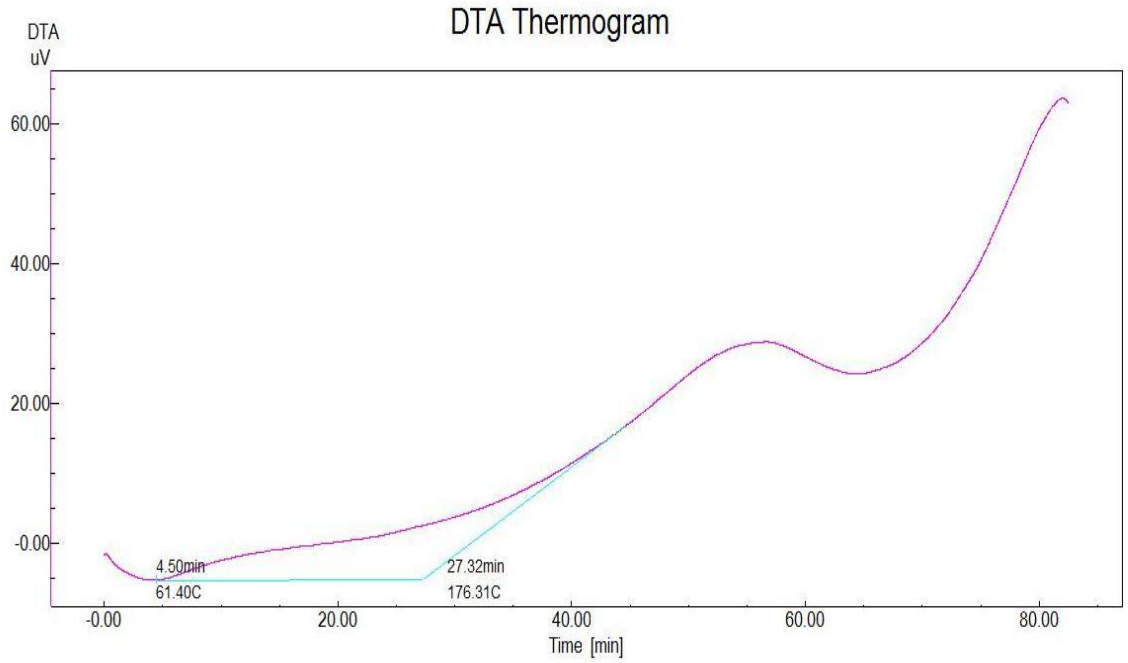


Fig. 4.23: DTA Thermogram for Sample SCCL 2

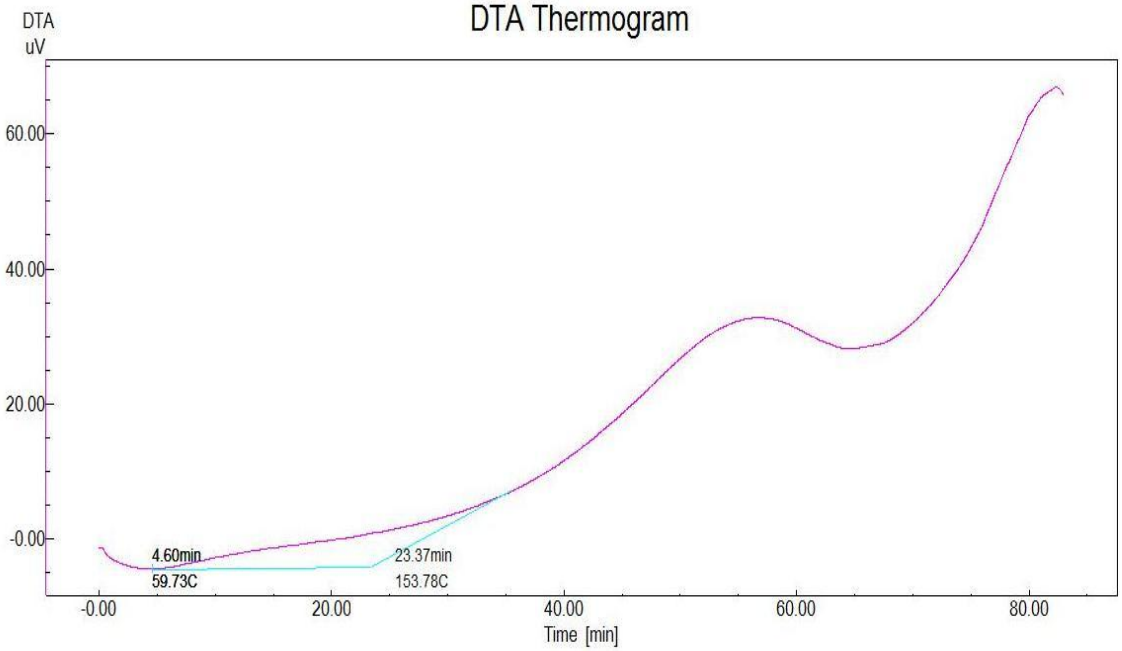


Fig. 4.24: DTA Thermogram for Sample SCCL 3

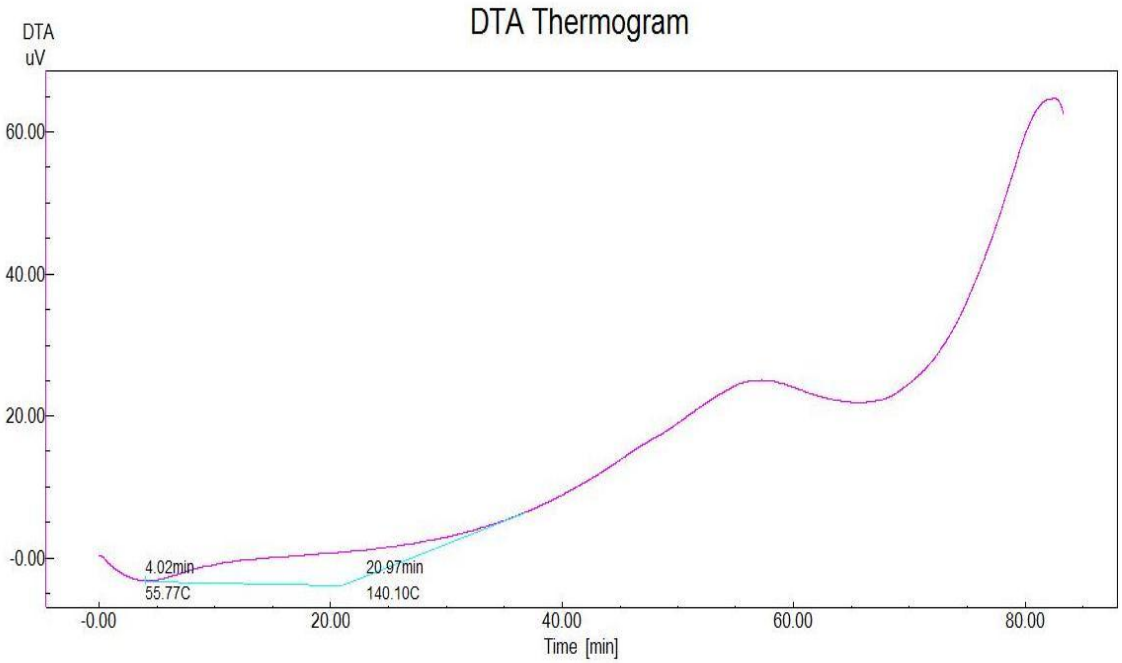


Fig. 4.25: DTA Thermogram for Sample SCCL 4

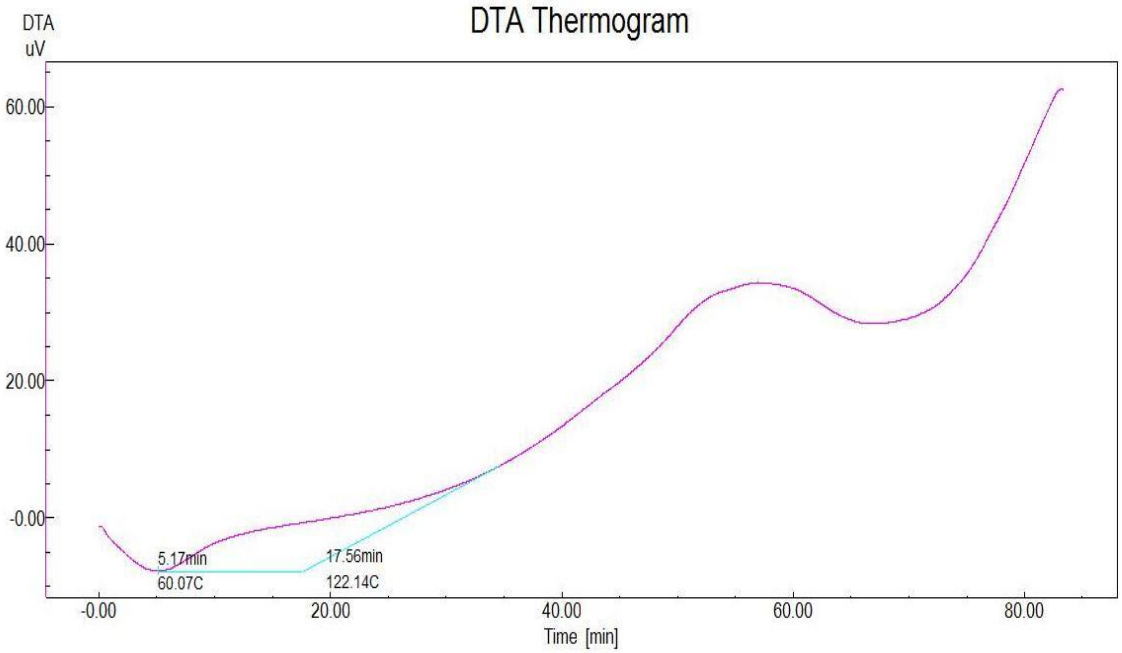


Fig. 4.26: DTA Thermogram for Sample SCCL 5

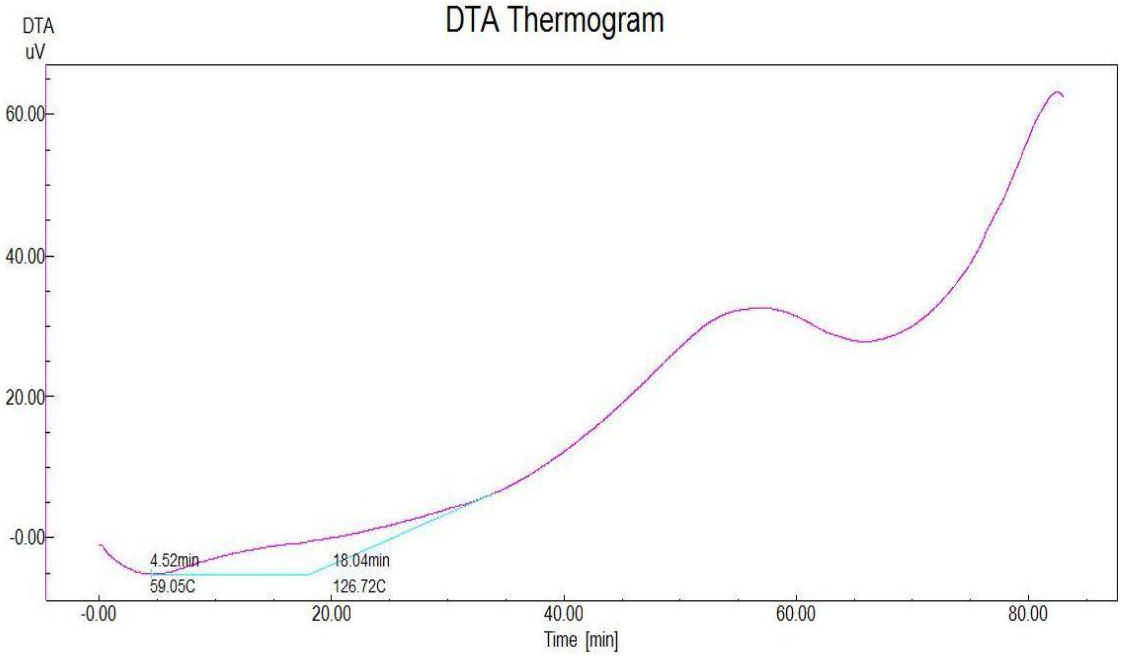


Fig. 4.27: DTA Thermogram for Sample SCCL 6

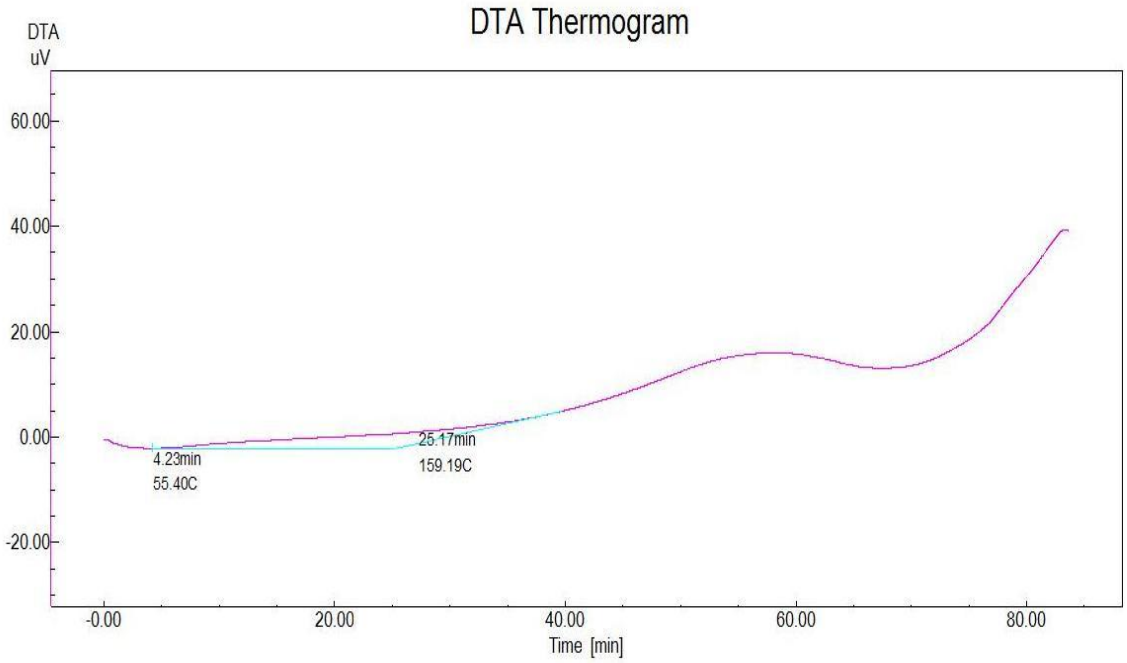


Fig. 4.28: DTA Thermogram for Sample SCCL 7

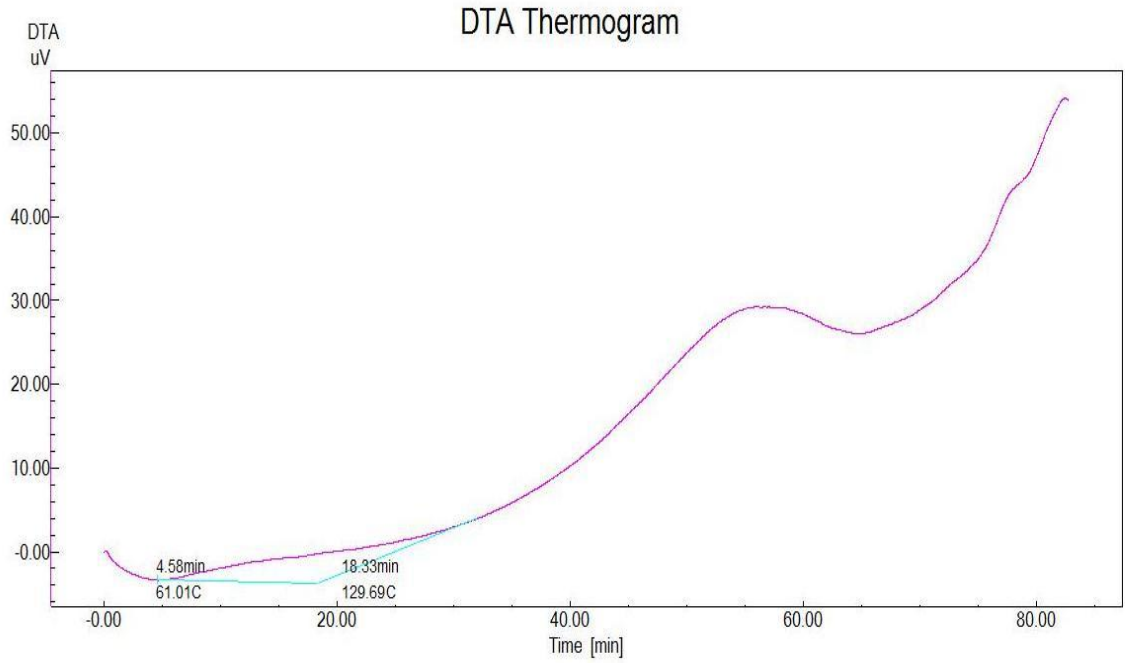


Fig. 4.29: DTA Thermogram for Sample SCCL 8

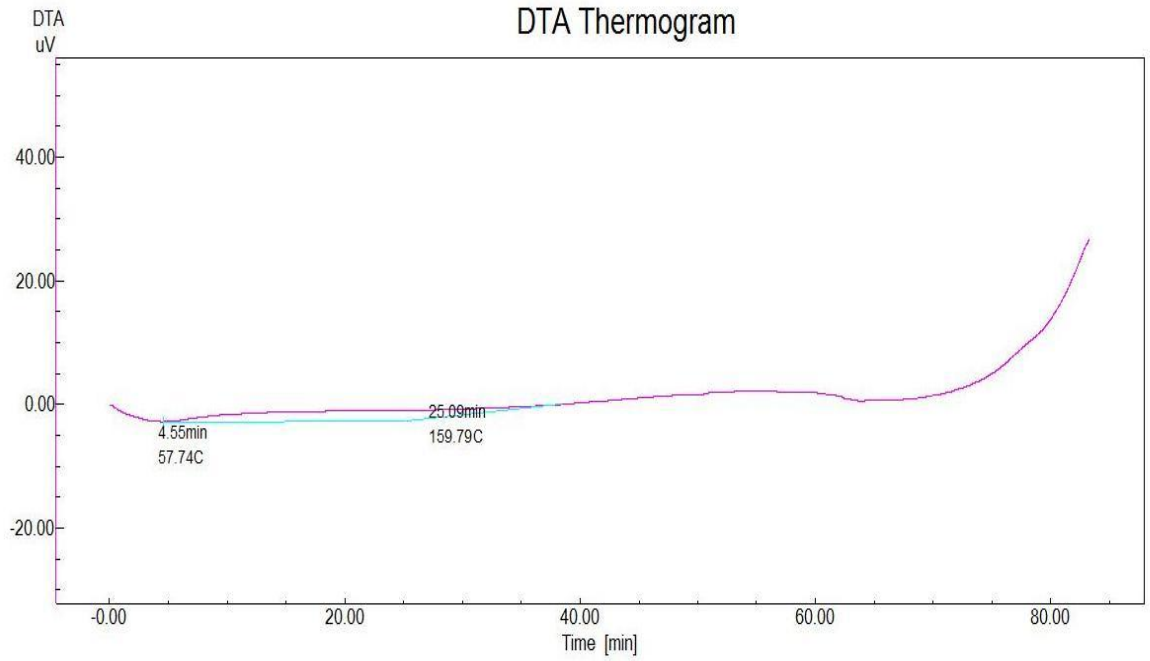


Fig. 4.30: DTA Thermogram for Sample SCCL 9

It has been shown by Banerjee and Chakravorty (1967) that a thermogram of coal can be divided into three segments or stages. These stages have also been identified in the thermograms generated from the results of the experiments as indicated in Figure 4.31. In the initial stage of heating (stage I), the endothermic reaction predominates, probably due to the release of inherent moisture in coal. In the second stage (stage II), the exothermic reaction becomes significant, but the rate of heat release is not steady all through, as it changes with temperature. A steep rise in heat evolution is observed in the third stage (stage III).

The rate of temperature rise in stage II is cited by different researchers, viz. Banerjee and Chakravorty (1967), Gouws and Wade (1989); as being less for coals with less susceptibility to spontaneous heating. The exothermicity in stage III is not regarded as a reliable indicator of the self heating risk, because it may be equally high for low rank coals. However, the temperature of transition or characteristic or onset temperature is considered to be significant. It is considered that in lower temperatures, the coal is more susceptible towards spontaneous heating.

Therefore, all the thermograms were analysed for the following details.

1. Onset temperature or characteristic temperature (T_c)

   The onset temperature or characteristic temperature was determined by the following steps:

   - A tangent was drawn at the inflexion point of the endothermic region and another at the rising portion of the curve of stage III.
   - The intersection between the two tangents gave the characteristic temperature.

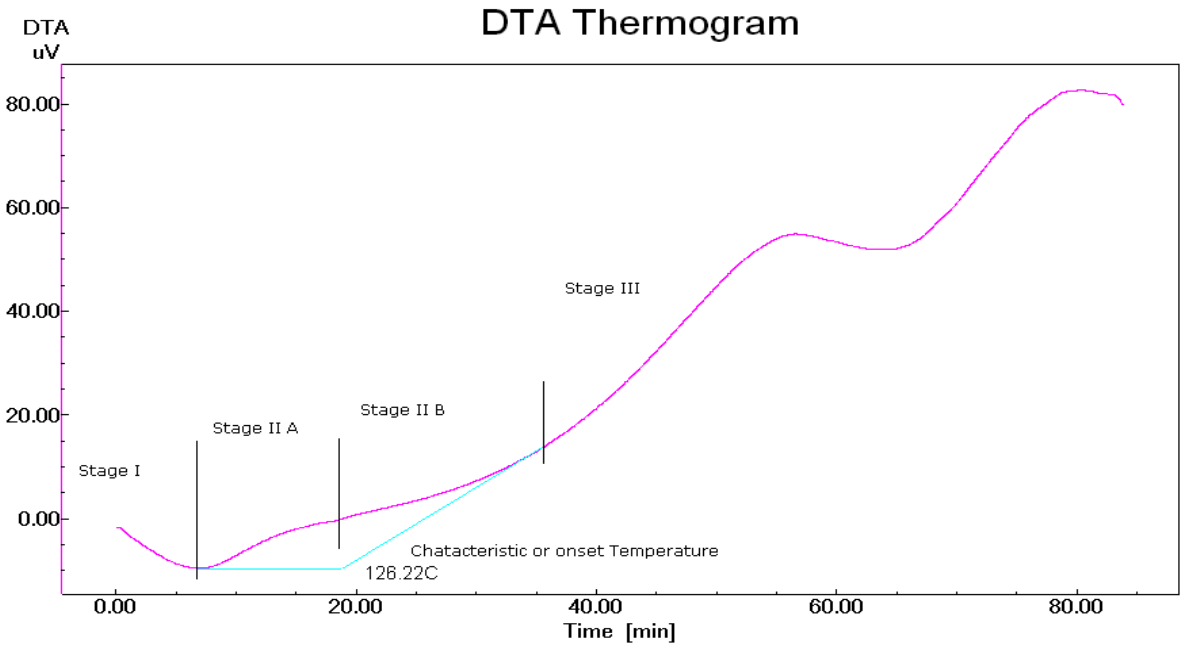


Fig 4.31: DTA Thermogram and Determination of Onset Temperature (T_c) for Sample MCL 1

2. Slopes of stage II of the thermogram

   In the thermograms obtained from the experiments, linear stage II exothermicity was not observed. In view of the disjointed nature of stage II slopes it was further divided into two different regions, viz. stage II A and stage II B. The following three parameters of stage II were determined for further analysis.

   (a) average slope of stage II A.

   (b) average slope of stage II B.

   (c) overall slope of stage II.

   The onset temperatures and slopes of II, II A and II B are presented in Table 4.5.

Table 4.5: Onset Temperature and Slopes of II, II A and II B obtained from DTA Thermograms

Sl. No.	Sample	Onset Temp., T_c (^{0}C)	Slope		
			II A	II B	II
1.	MCL1	179.57	0.0747	0.1515	0.0979
2.	MCL2	164.8	0.1228	0.2064	0.1354
3.	MCL3	166.23	0.1015	0.1866	0.1137
4.	MCL4	166.22	0.1205	0.231	0.1364
5.	MCL5	157.49	0.0958	0.1619	0.1084
6.	MCL6	170.9	0.0308	0.0929	0.0499
7.	MCL7	140.73	0.1008	0.1729	0.1182
8.	MCL8	155.93	0.0122	0.0605	0.0211
9.	MCL9	147.29	0.016	0.0753	0.0288
10.	MCL10	158.46	0.0145	0.0743	0.0222
11.	MCL11	153.34	0.0177	0.066	0.0273
12.	MCL12	124.82	0.0229	0.0681	0.0295
13.	MCL13	171.87	0.0167	0.0485	0.0216
14.	SCCL1	144.98	0.0533	0.078	0.0565
15.	SCCL2	176.31	0.0615	0.1392	0.0836
16.	SCCL3	153.78	0.056	0.1245	0.0744
17.	SCCL4	141.1	0.0445	0.0932	0.0562
18.	SCCL5	122.14	0.1021	0.1076	0.0885
19.	SCCL6	126.72	0.0712	0.0909	0.0732
20.	SCCL7	159.19	0.0272	0.0605	0.0373
21.	SCCL8	127.67	0.0473	0.079	0.056
22.	SCCL9	159.79	0.0334	0.012	0.0128

4.6. Wet Oxidation Potential Analysis

The coal molecule may be considered as consisting of two parts: the aliphatic or hydro-aromatic structure that are more prone to oxidation and condensed aromatic structure, which are resistant to oxidation. Presence of hydroxyl group in the aromatic structure part gives a very high degree of reactivity to coal structure and they get oxidised faster. Thus, low rank coals are rather easily oxidised due to the above and also due to smaller degree of condensation of aromatic structures in them. Lower rank coals on oxidation produce large amounts of aliphatic acids compared to higher rank coals, because low rank coals contain more branched aliphatic hydro-carbons. Since the high rank coals have structure close to that

of graphite, it is less liable to oxidation and the products contain more aromatics than aliphatics. It is generally seen that lower the potential difference, lower is the susceptibility of coal to spontaneous heating.

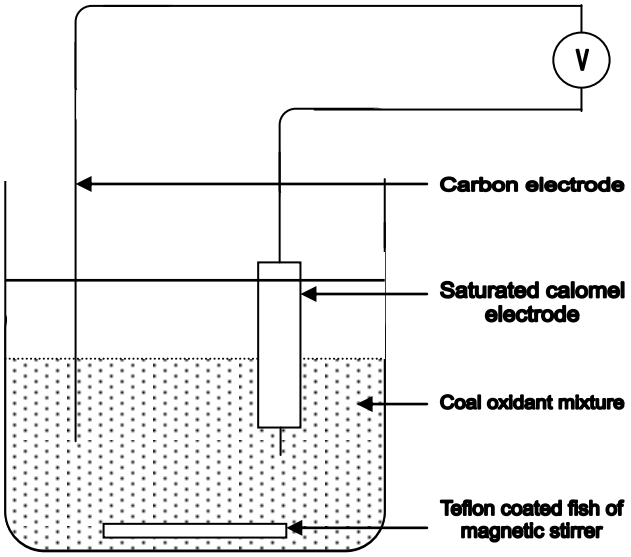
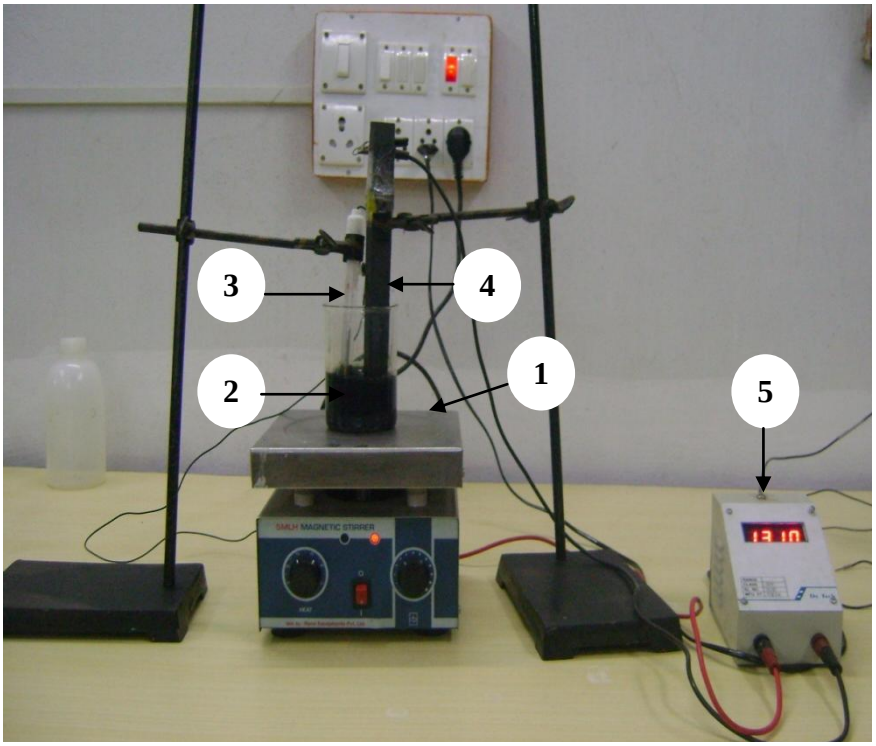


Fig 4.32: Wet Oxidation Apparatus

Experimental Procedure

The beaker along with the electrodes (Figs. 4.32 and 4.33)) was placed over a magnetic stirrer such that a homogeneous mixture of coal and alkali solution is maintained. The teflon coated fish of the magnetic stirrer was placed inside the beaker. 0.5 g of coal sample of –212μ (micron) size was mixed with 100 ml of decinormal solution of potassium permanganate ($KMnO_4$) in 1N potassium hydroxide (KOH) solution in a beaker and the coal sample was subjected to wet oxidation process. The coal-oxidant suspension was continuously stirred using the magnetic stirrer. The potential difference (EMF) was recorded between the calomel and carbon electrodes over a period of time by using a millivoltmeter till the potential difference attained a nearly constant value. The graphs between Time vs. EMF (millivolt) for all the samples are plotted in Figs. 4.34 to 4.38. Different samples require different time duration for attaining a nearly constant potential difference (ΔE). It was observed that almost all the coal samples attained a constant value after 25 minutes. Therefore this time period was chosen to find out the wet oxidation potential difference for a comparative study. The results are presented in Table 4.6.



1. Magnetic Stirrer
2. $KMnO_4$, KOH and Coal Solution
3. Calomel Electrode
4. Carbon Electrode
5. Millivoltmeter

Fig. 4.33: Experimental Set up of Wet Oxidation Apparatus

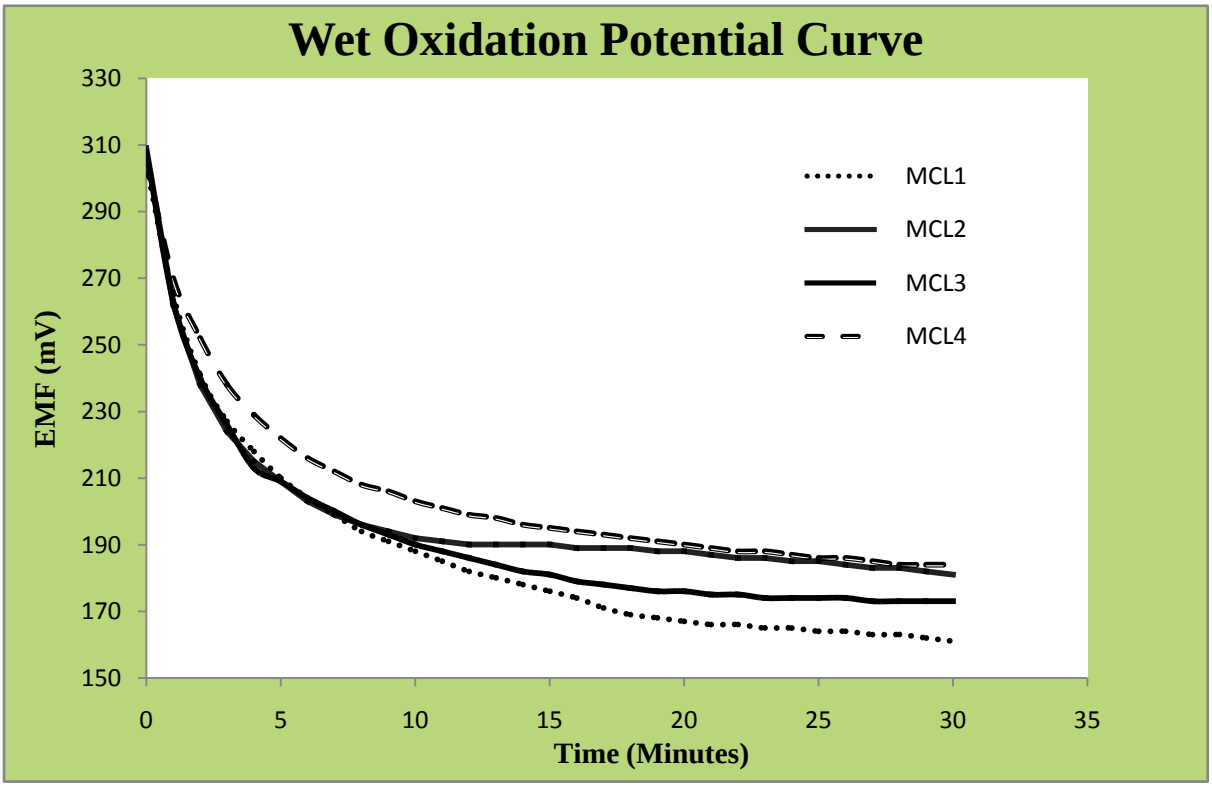


Fig. 4.34: Wet Oxidation Curves for Samples MCL 1-4

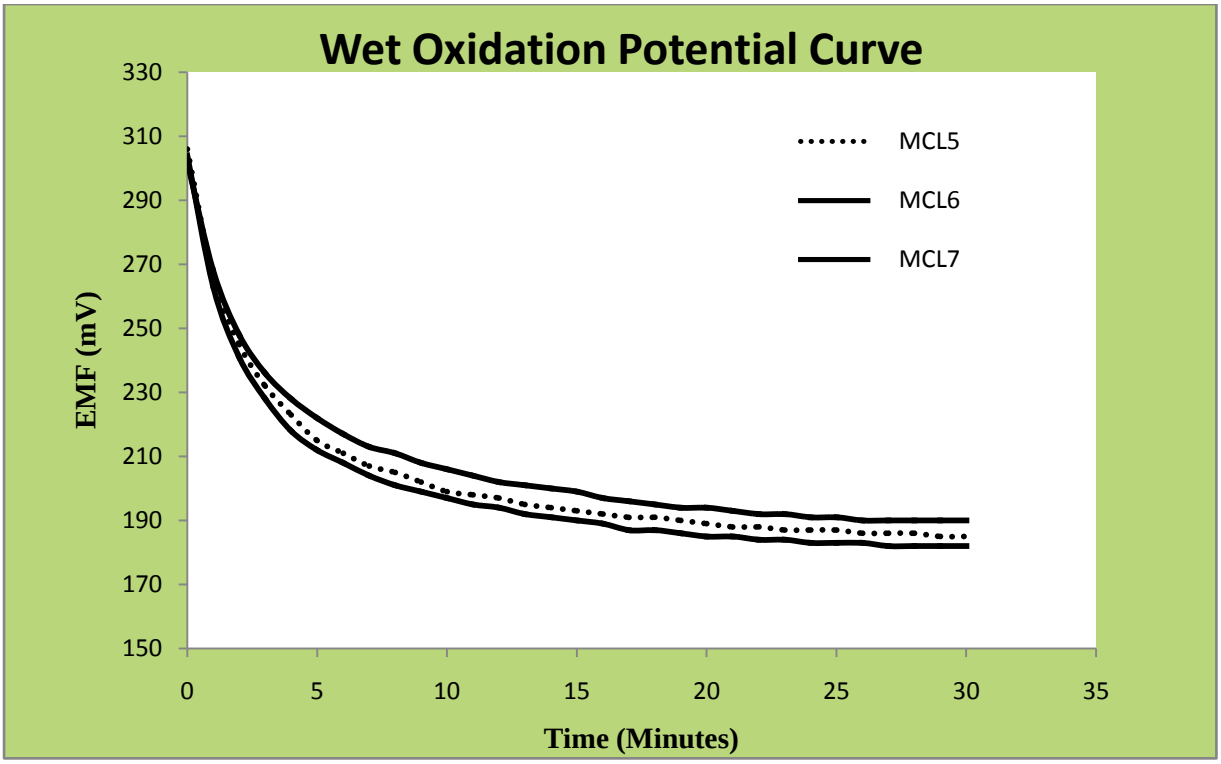


Fig. 4.35: Wet Oxidation Curves for Samples MCL 5-7

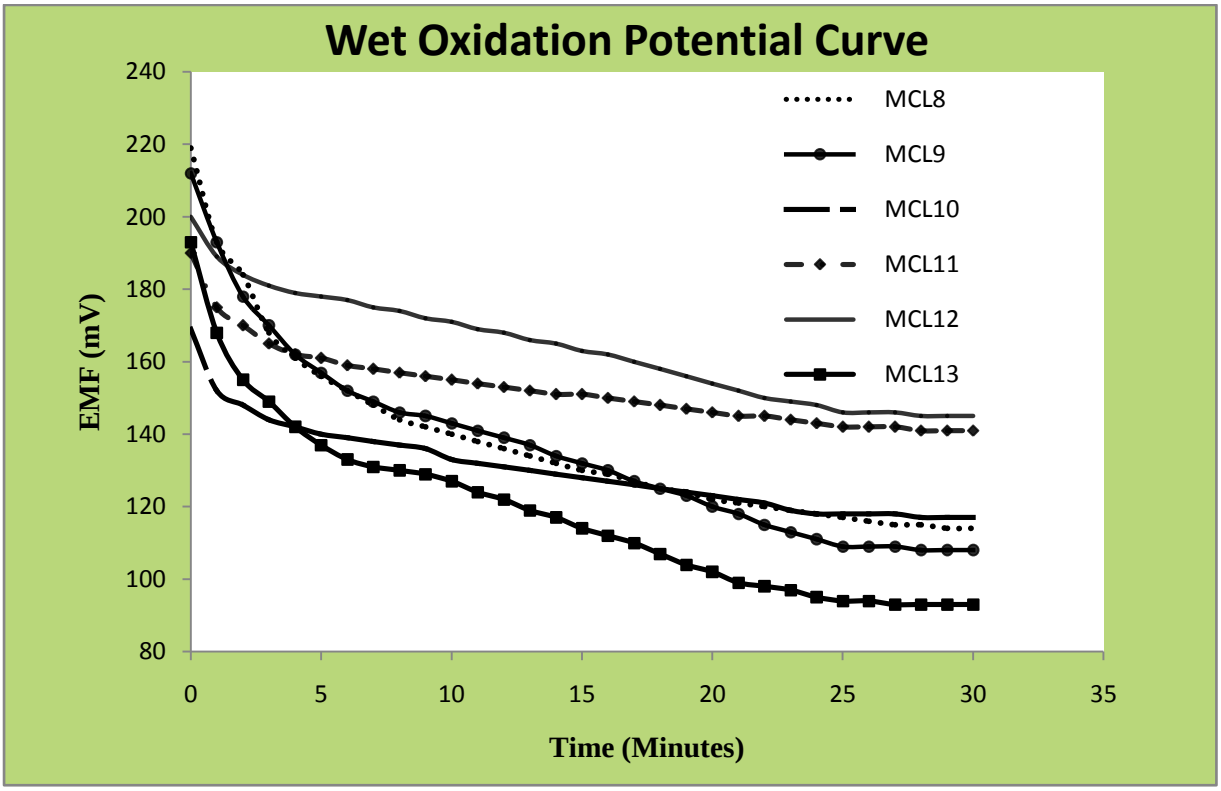


Fig. 4.36: Wet Oxidation Curves for Samples MCL 8-13

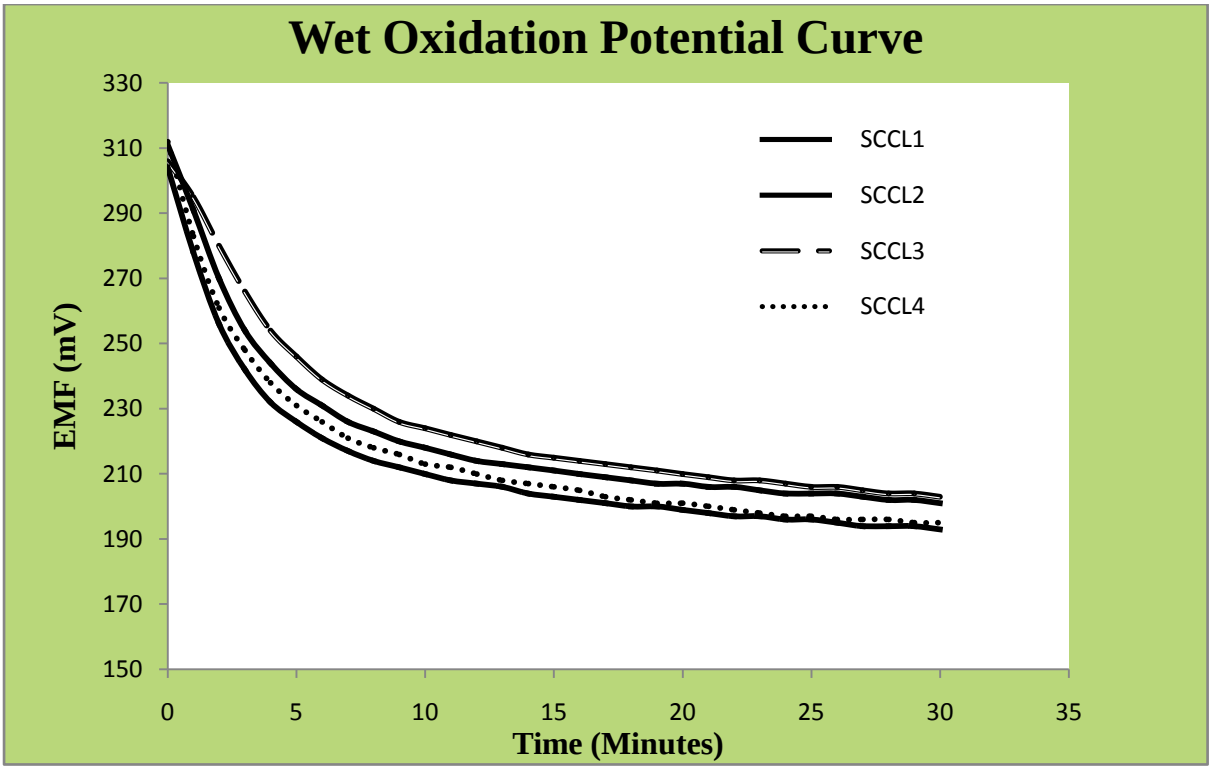


Fig. 4.37: Wet Oxidation Curves for Samples SCCL 1-3

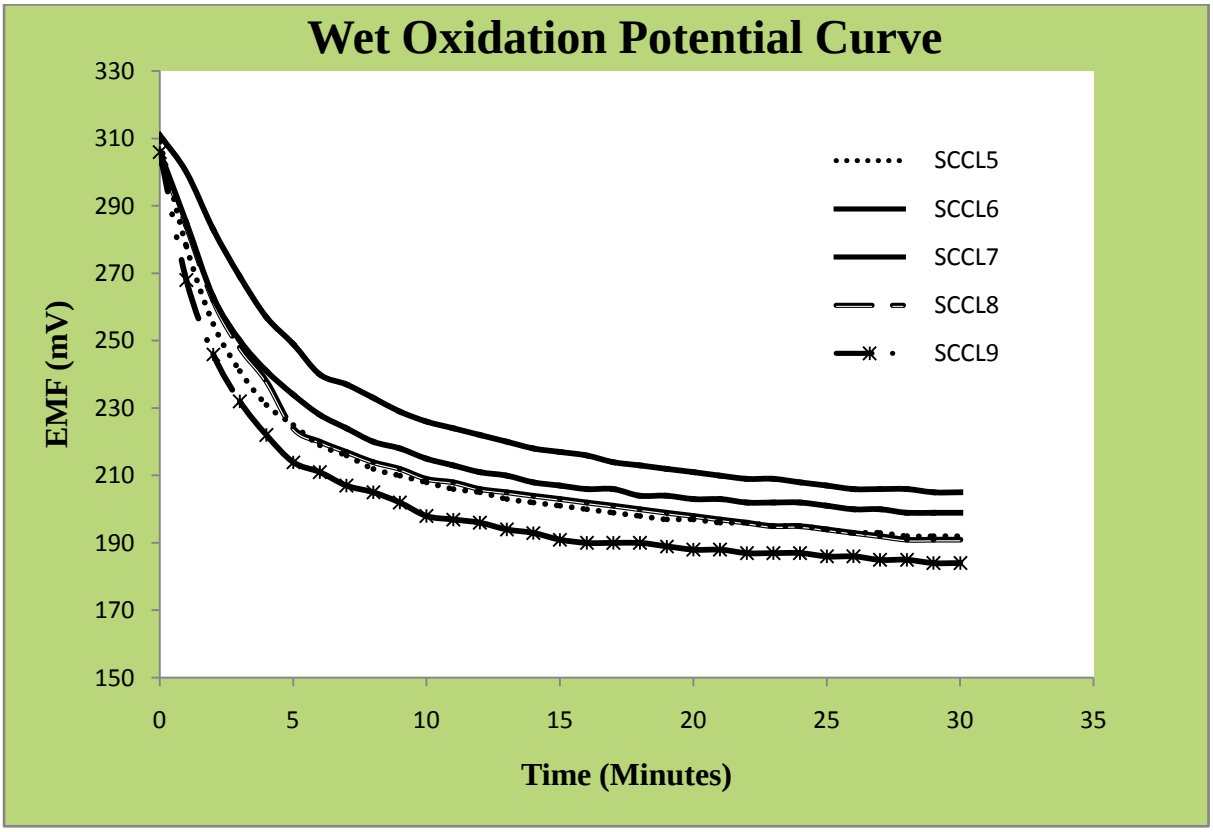


Fig. 4.38.: Wet Oxidation Curves for Samples SCCL 5-9

Table 4.6: Wet Oxidation Potential Difference of the Coal Samples

Sl. No.	Sample	Wet Oxidation Potential Difference, (mV)
1.	MCL1	140
2.	MCL2	124
3.	MCL3	134
4.	MCL4	119
5.	MCL5	119
6.	MCL6	121
7.	MCL7	111
8.	MCL8	102
9.	MCL9	103
10.	MCL10	51
11.	MCL11	48
12.	MCL12	54
13.	MCL13	99
14.	SCCL1	107
15.	SCCL2	108
16.	SCCL3	100
17.	SCCL4	115
18.	SCCL5	115
19.	SCCL6	105
20.	SCCL7	104
21.	SCCL8	110
22.	SCCL9	120

Chapter 5

DISCUSSION AND CONCLUSION

.

Discussion

Conclusion

5.1. Discussion

In all 22 coal samples were collected from different mines of MCL and SCCL and assessed for spontaneous heating susceptibility. 13 were from Talcher and Ib valley Coal fields and 9 from Godavari Valley Coal field (Srirampur Area)

The Moisture content of coal varied from 2.149 (Sample SCCL 3) to 14.5 (Sample MCL 10). Coal from Talcher and Ib Valley areas had comparatively higher moisture content compared to the ones from Srirampur Area (Godavari Valley Coalfield), SCCL. It was seen that samples of higher moisture content are more prone to spontaneous heating.

It may be observed from Table 5.1, the Volatile Matter Content of Coal varied from 20.007 (Sample SCCL9) to 40.677 (Sample SCCL 7). The Gross Calorific Value of Coal Varied from 3130.98 (Sample MCL 1) to 6693.61 (Sample SCCL 8). The calorific values of coals from Godavari Valley Coal field were found to be higher.

The Ash content varied from 7.68 (Sample MCL 12) to 52.3 (Sample MCL 9). Some coals belonging to Talcher and Ib Valley areas had notably high ash content.

The Crossing Point temperature of coal varied from 154(Sample MCL 1) to 183 (Sample SCCL 8) with 19 samples having values ranging from 160 -183. This indicated that a majority of them were not susceptible to spontaneous combustion.

It may be observed from Table 5.2, transition temperature of coal varied from 122.14 (Sample SCCL 5) to 179.57 (Sample MCL 1). It was seen that the higher the transition temperature, lower is the spontaneous heating susceptibility of coal seams. The II A slope values varied from 0.1228 (Sample MCL 8) to 0.1228 (Sample MCL 2). II B slope values varied from 0.012 (Sample SCCL 9) to 0.231 MCL 4. II slope values varied from 0.0128 (SCCL 9) to 0.1364 (Sample MCL 4). It has been observed in the past that higher the slope value, higher is the spontaneous heating susceptibility of coal. Sample MCL 7 had one of the lowest onset temperature and a high slope value among all the samples. So it may be considered to be highly susceptible to spontaneous heating. This sample belongs to the Talcher coalfields and it is corroborated from the fact that it is known to be a fiery seam in the field. Panigrahi and Sahu (2004) have found that the coal seams having onset temperature in the range of 122^0C to 140^0C are highly susceptible to heating. Therefore, Samples MCL 7, 12 and SCCL 5, 6 and 8 could be considered as moderately susceptible to heating.

The Wet Oxidation Potential Difference varied from 48 (Sample MCL 11) to (Sample MCL 1) 140. Higher the difference of Wet oxidation potential more is the susceptibility to spontaneous combustion. Coals belonging to Godavari Valley Area had less value indicating that it was less susceptible to spontaneous combustion whereas coals belonging to Talcher and Ib Valley areas had high values indicating that it was prone to spontaneous heating.

Correlation studies were carried out between the different susceptibility indices and the coal characteristics as obtained from proximate analysis. The susceptibility indices were taken as dependent variables and each constituent obtained from the proximate analysis and GCV as an independent variable. The correlation coefficients obtained in all cases are presented in Table 5.1.

Table 5.1: Correlation coefficients between different susceptibility indices and proximate analysis and GCV

Sl. No.	Independent Variable	Dependant Variable	Correlation Coefficient, r
1.	Moisture	CPT	0.217
2.	Volatile Matter	CPT	-0.8
3.	Ash	CPT	-0.227
5.	GCV	CPT	0.263
6.	Moisture	T_c	0.23
7.	Volatile Matter	T_c	-0.221
9.	Ash	T_c	0.437
10.	GCV	T_c	-0.512
11.	Moisture	II	0.063
12.	Volatile Matter	II	-0.042
13.	Ash	II	-0.063
14.	GCV	II	0.067
15.	Moisture	II A	0.152
16	Volatile Matter	II A	0.005
17.	Ash	II A	0.097
18.	GCV	II A	-0.031
19.	Moisture	II B	0.063
20.	Volatile Matter	II B	0.035
21.	Ash	II B	0.017
22.	GCV	II B	0.043

It may be observed from the aforementioned correlation study that

- Crossing Point Temperature (CPT) values show better correlation with Volatile Matter.
- Transition temperature (Tc) obtained from DTA thermogram shows better correlation with Ash and GCV of proximate analysis than other constituents.

5.2 Conclusion

From the aforementioned discussion, it may be concluded that samples MCL 1, 5, 7, 12 and SCCL 5 and 8 are moderately prone to spontaneous heating. Rest of the samples are less susceptible to spontaneous heating.

Crossing point temperature is taken as the measure of spontaneous heating susceptibility of coals in India. However, it has been observed in the past that it does not predict the susceptibility for all types of coals, particularly for high moisture coals. Moreover, it takes approximately 3 hours to determine the CPT which is time consuming. In the event of a power failure, the whole experiment has to be repeated. Reproducibility of experimental results of Crossing Point Temperature also posed a major problem in the dissertation.

Some of these demerits can be overcome by carrying out the differential thermal analysis, where it takes about 2 hours for the completion of an experiment. The results are highly reproducible and it is very easy to determine the transition temperature and slope values using various statistical packages.

The wet oxidation potential difference may be taken as another indicator of spontaneous heating. Coal samples with high susceptibility to spontaneous heating will have higher potential difference values. This experiment is very easy to perform and takes only thirty minutes to complete. However, no clear cut guideline has been established till date to standardise the wet oxidation potential difference values with respect to their spontaneous heating susceptibility.

Chapter 6

REFERENCES

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6. REFERENCES

Banerjee, S. C., Chakravorty, R. N. , 1967, Use of DTA in the study of spontaneous combustion of coal, Journal of Mines, Metals and Fuels, January, pp. 1-5.

Banerjee, S. C., Nandy, D. K. , Banerjee, D. D., Chakravorty, R. N., 1972, Classification of coal with respect to their Susceptibility to Spontaneous Combustion, MGMI, July, Vol. 69, No. 2, pp. 15-28.

Banerjee, S. C., 1985, Spontaneous Combustion of Coal and Mine Fires, Oxford & IBH Publishing Co. Pvt. Ltd., 1st Ed., pp. 1-38.

Feng, K. K., Chakravarty, R. N., Cochrane, T. S., 1973, Spontaneous combustion – a coal mining hazard, CIM Bulletin. Oct., pp. 75-84.

Gouws, M.J., Wade, L., 1989, The self-heating liability of coal: Prediction based on composite indices, Mining Science and Technology. 9, pp. 81-85.

I.S. (Indian Standard): 1350 (Part-I) – 1984, Methods of Test for Coal and Coke: Proximate Analysis, Bureau of Indian Standards, New Delhi, pp. 3-28.

I.S. (Indian Standard): 1350 (Part II) - 2000, Methods of Test for Coal and Coke: Determination of Calorific Value, Bureau of Indian Standards, New Delhi, pp. 3-24.

Mahadevan, V., Ramlu, M. A., 1985, Fire risk rating of coal mines due to spontaneous heating, Journal of Mines, Metals and Fuels. August, pp. 357-362.

Nandy, D. K., Banerjee, D. D., Chakravorty, R.N., 1972, Application of crossing point temperature for determining the spontaneous heating characteristics of coal, Journal of Mines, Metals and Fuels, pp. 20-41.

Nordon, P., Young, B. C. and Bainbridge, N.W., 1979, The rate of oxidation of char and coal in relation to their tendency to self-heat, Fuel, Vol. 58, p. 443-449.

Ramlu, M.A., 1997, Mine Disasters and Mine Rescue, Oxford & IBH Publishing Co. Pvt. Ltd., 1st Ed., pp. 1-19.

Oliveira, A., 1998, A digital differential thermal analysis instrument, Measurement, Volume 23, Issue 1, pp. 48.

Panigrahi et al, 2001, A study of susceptibility of Indian coals to spontaneous combustion and its correlation with their intrinsic properties, Mine Environment and Ventilation, Oxford & IBH Publishing Co., New Delhi, pp. 247-254.

Panigrahi, D. C., Udaybhanu, G., Ojha, A., 1996, A comparative study of wet oxidation method and crossing point temperature method for determining the susceptibility of Indian coals to spontaneous heating, Proceedings of Seminar on Prevention and Control of Mine and Industrial Fires – Trends and Challenges. Calcutta. India. Dec., pp. 101-107.

Panigrahi D.C., Alok Ojha, Saxena N.C. and. Kejriwal B.K , 1997, A study of coal oxygen interaction by using Russian U-Index and its correlation with basic constituents of coal with particular reference to Jharia coal field, 27th international conference of safety in mines research institutes, New Delhi ,India, pp. 493-499.

Sahu, H. B., Panigrahi, D. C. and Mohapatra, S. S., 2009, Classification of coal seams with respect to their spontaneous heating susceptibility using K-Means Clustering, Proceedings of the Ninth International Mine Ventilation Congress, New Delhi, pp. 273-281.

Sahu, H. B., Mohapatra, S. S. and Panigrahi, D. C., 2009, An empirical approach for classification of coal seams with respect to the spontaneous heating susceptibility of Indian coals, International Journal of Coal Geology, vol. 80, pp. 175-179.

Tarafdar, M. N., Guha, D., 1989, Application of wet oxidation processes for the assessment of the spontaneous heating of coal, Fuel. 68, March, pp. 315-317.